

Stress Corrosion Cracking Response of 304 Stainless Steel in Aerated and Deaerated Water

W. J. Mills

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ABSTRACT

Scoping stress corrosion cracking (SCC) tests of 304 stainless steel (SS) were performed in 75°C and 250°C aerated pressurized water (APW) and 250°C deaerated pressurized water (DPW). The 250°C APW environment was used to initiate intergranular stress corrosion cracking (IGSCC) and then the water was deaerated and hydrogenated to see if IGSCC continued in 250°C DPW. Tests were performed with and without 200 ppb $\text{SO}_4^{=}$. The 304 SS test materials were evaluated in either the as-received, heavily sensitized (649°C for 1 h), fully sensitized (1099°C for 1 h / water quench / 621°C for 17 h) or 20% cold rolled condition. At the beginning of each test sequence, specimens were subjected to continuous cycling with a 500s rise / 500s fall or a 5000s rise / 500s fall to promote the transition from a transgranular (TG) precrack to an IG crack. After generating a uniform crack under continuous cycling conditions, a trapezoidal waveform with 500s rise / 9000s hold / 500s fall was used to characterize the SCC behavior. Crack growth rates (CGRs) were monitored continuously with the electric potential drop (EPD) method and were corrected based on physical crack length measurements obtained when specimens were destructively evaluated.

Continuous cycling with a 500s or 5000s rise time was found to produce both TG faceting and IGSCC in fully sensitized 304 SS tested in 75°C APW with 7 ppm O_2 and 200 ppb $\text{SO}_4^{=}$. However, no measurable crack extension occurred when a 9000 s hold time was introduced. Extensive IGSCC occurred in heavily sensitized and fully sensitized 304 SS in 250°C APW with 1 ppm O_2 and 200 ppb $\text{SO}_4^{=}$. IGSCC initiated under continuous cycling conditions with a 500 s rise time, and rapid IGSCC occurred when a 9000 s hold time was introduced. During the trapezoidal waveform test with a 9000 s hold, CGRs ranged from 1 to 3 mils/day for the heavily sensitized material and 5 to 10 mils/day for the fully sensitized material. When the test environment was deaerated and hydrogenated to 30 cc H_2 /kg H_2O , CGRs decreased by an order of magnitude (~0.1 mils/day), and the cracking mode in 250°C DPW was predominantly TG. The only material that exhibited evidence of IGSCC in 250°C DPW was 20% cold rolled 304 SS. IGSCC readily initiated in 250°C APW and continued in 250°C DPW. Metallographic and fractographic examinations showed that the IG cracking was highly branched and discontinuous.

Results from the scoping SCC tests have revealed significant information regarding the nature of IG and TG cracking of 304 SS in APW and DPW. The key finding is that cold work appears to be more important than sensitization in producing IGSCC in DPW.



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Bechtel Bettis
Plant Materials Technology



SCC Response of 304 Stainless Steel in APW & DPW

PURPOSE

Scoping SCC tests of 304 SS were performed in 75°C & 250°C aerated pressurized water (APW) & 250°C deaerated pressurized water (DPW) to determine the conditions required to cause SCC.

APPROACH

Scoping SCC tests of 304 SS in either the sensitized or CW^{ed} condition were performed in:

- 75°C APW.
- 250°C APW / 250°C DPW

250°C APW was used to initiate IGSCC.

Water was degassed & hydrogenated to see if SCC continued in DPW.



EXPERIMENTAL PROCEDURE

Test Materials (304 SS Heats F0313, 61115)

- As-Received Condition
- Heavily Sensitized 649°C for 1 h
- Fully Sensitized 1099°C for 1 h / Water Quench / 621°C for 17 h
- Cold Rolled 20%

Test Methodology

- SCC tests performed on fatigue precracked 1T or 1.9T CT or 1T WOL
- Continuous cycling with 500s rise / 500s fall or 5000s rise / 500s fall promote TG / IG transition
- Trapezoidal waveform with 500s rise / 9000s hold / 500s fall.
- EPD method, corrected based on DE results, used to compute CGRs.

Test Environments

- 75°C APW with 7 ppm O₂ & 200 ppb SO₄.
- 250°C APW with 1 ppm O₂ with & without 200 ppb SO₄.
- 250°C DPW with 25-30 cc H₂/kg H₂O with 200 ppb SO₄.



CONCLUSIONS FROM SCC SCOPING TESTS

SCC Scoping Tests revealed significant information regarding IG & TG cracking in APW & DPW;

important in providing direction for future testing

Tests on sensitized 304 SS (non-CW^{ed}) produced IGSCC in 250°C APW & TGSCC in 250°C DPW.

Tests on sensitized 304 SS (non-CW^{ed}) in 75°C APW

produced both IGSCC and TG fatigue during continuous cycling,
but produced no measurable SCC in 75°C APW with a 9000s hold.

20% CW 304 SS was the only material that produced IGSCC in DPW.

CW appears to be more important than sensitization in producing IGSCC in 250°C DPW.



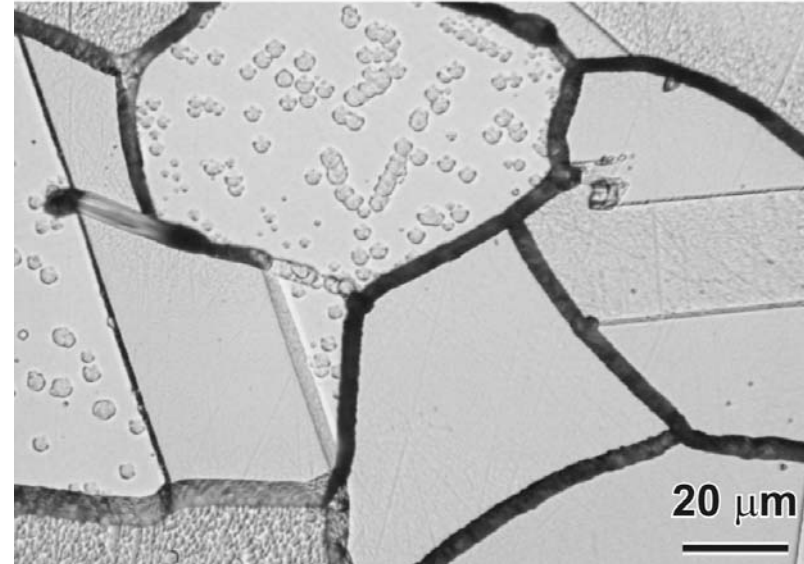
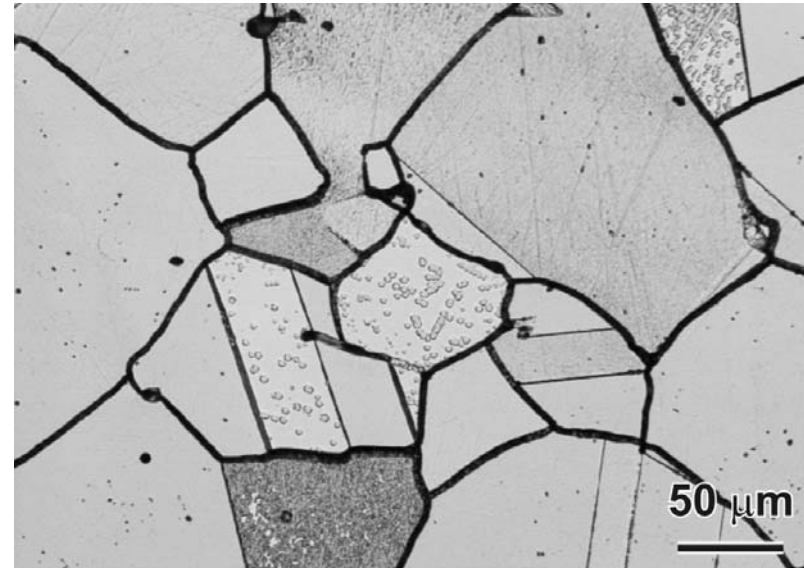
**Fully Sensitized 304 SS Heat 61115
(CT-16P)**

1099°C for 1 h

Water Quench

621°C for 17 h

Continuous network of ditched GBs.





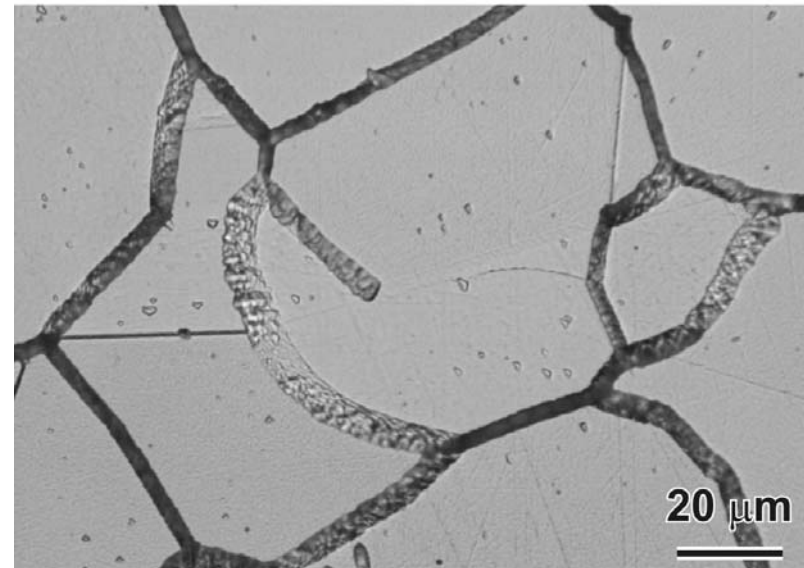
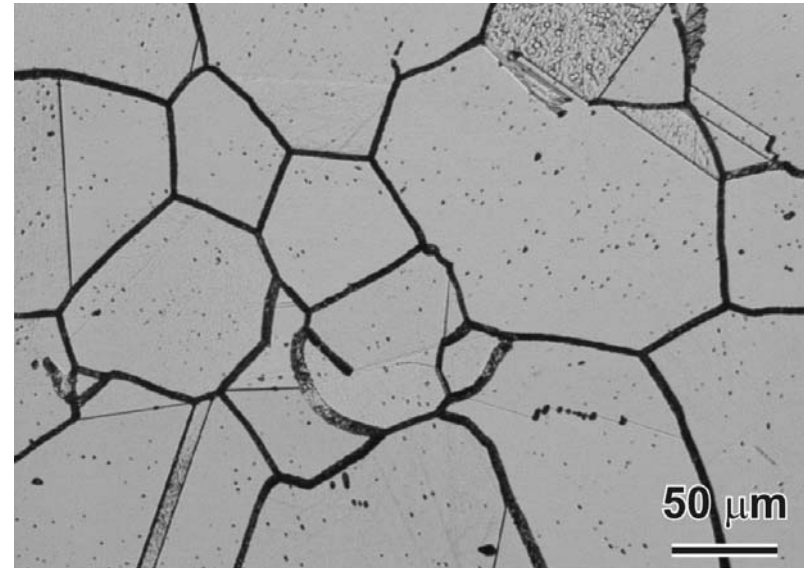
**Fully Sensitized 304 SS Heat F0313
(WLP2-10)**

1099°C for 1 h

Water Quench

621°C for 17 h

Continuous network of ditched GBs.

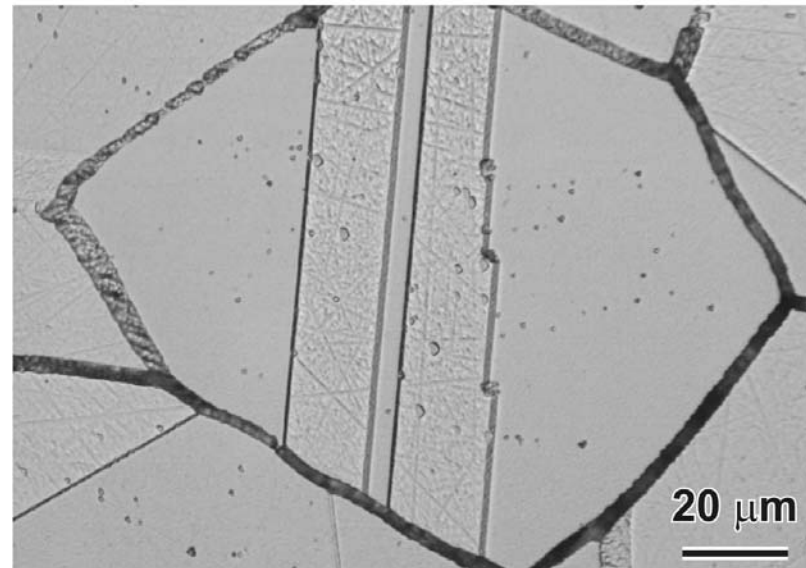
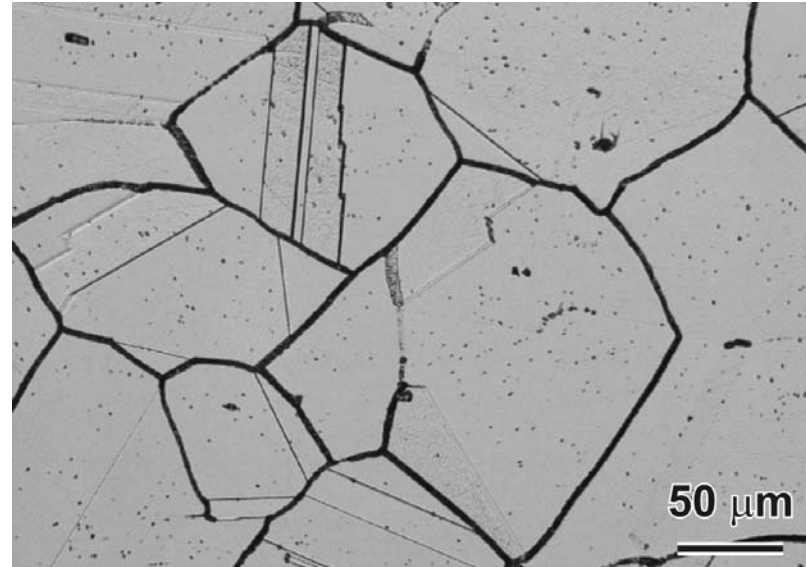




**Heavily Sensitized 304 SS Heat F0313
(WLP2-4, WLP2-6)**

649°C for 1 h

Most grains are surrounded by ditched
GBs.

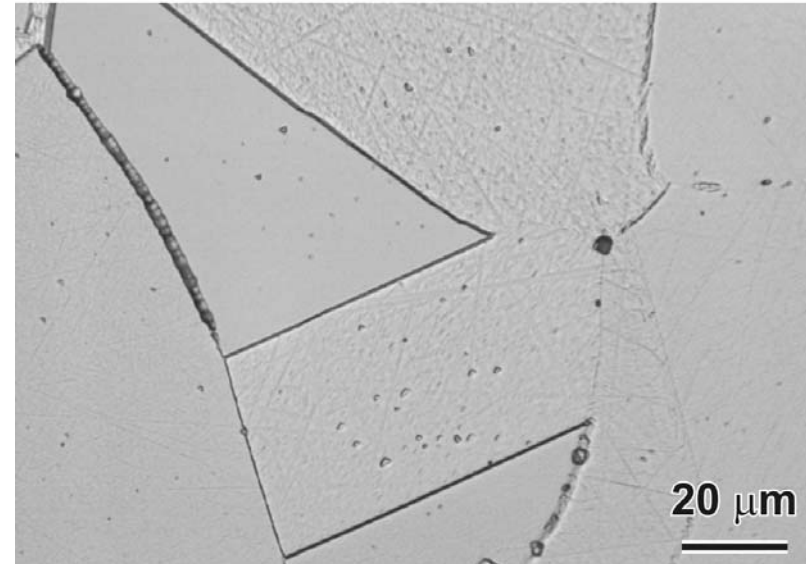
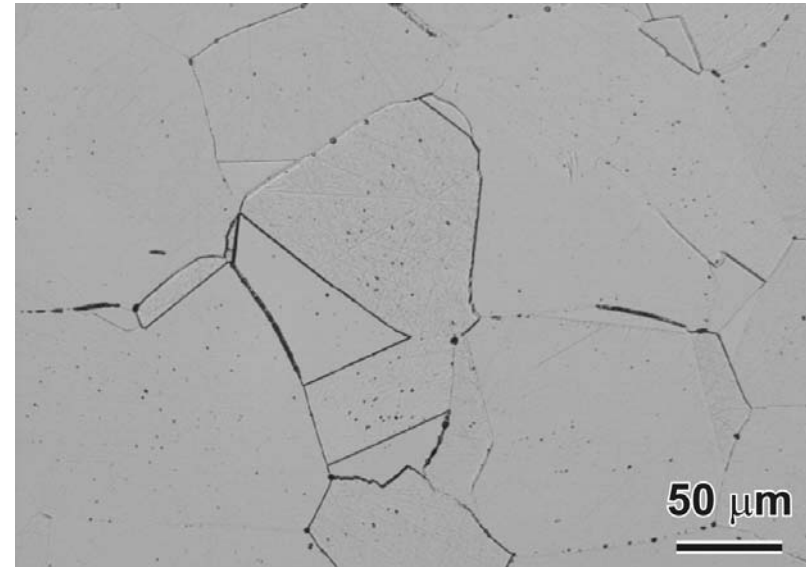




**As-Received 304 SS Heat F0313
(WLP2-8)**

Dual structure.

~10% of GBs exhibit a ditched structure.





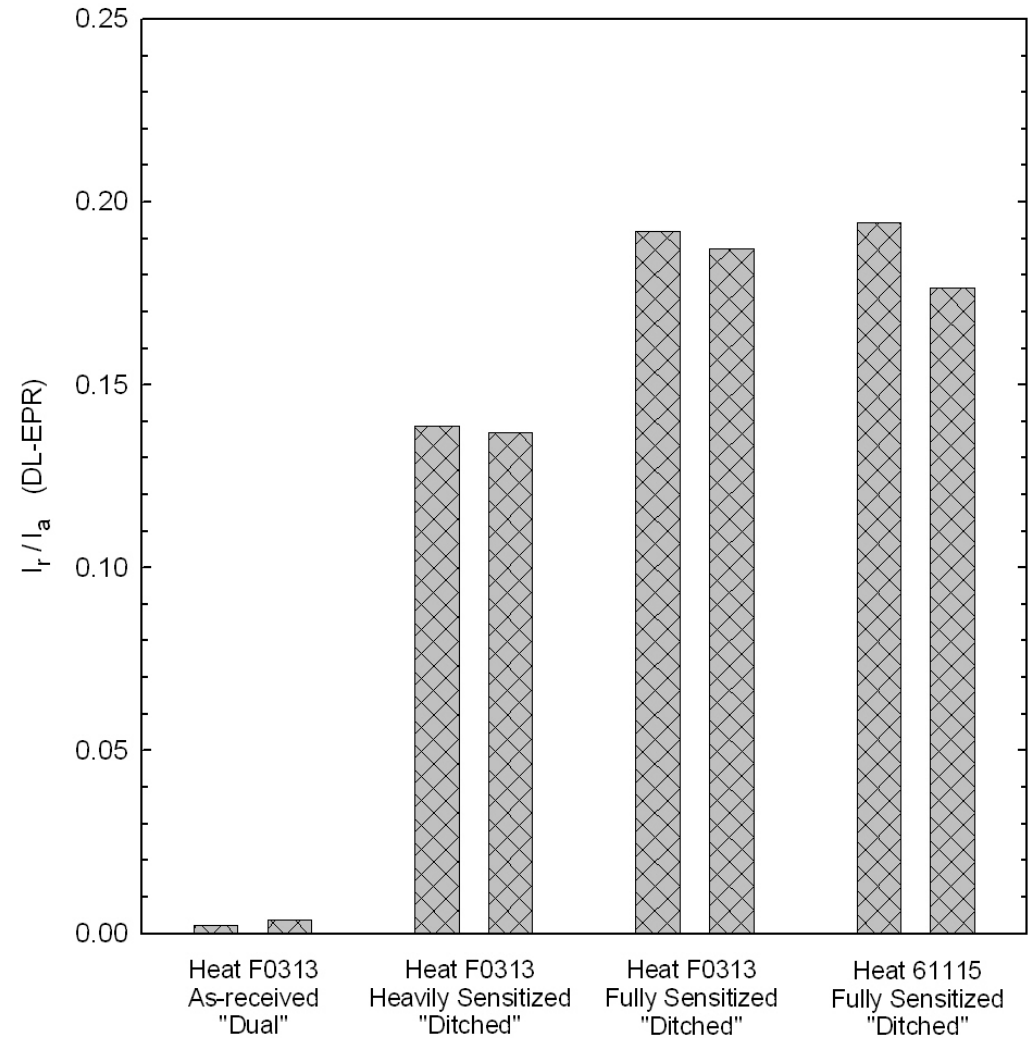
Double Loop-EPR Testing

Heavily and Fully Sensitized 304 SS exhibited very high I_r/I_a values (consistent with ditched structure).

Sensitized materials should be very susceptible to IGSCC.

As-Received "dual" 304 SS exhibited very low I_r/I_a values.

As-received material should resist IGSCC.





Fully Sensitized 304 SS
Tested in 75°C APW with 7 ppm O₂ & 200 ppb SO₄

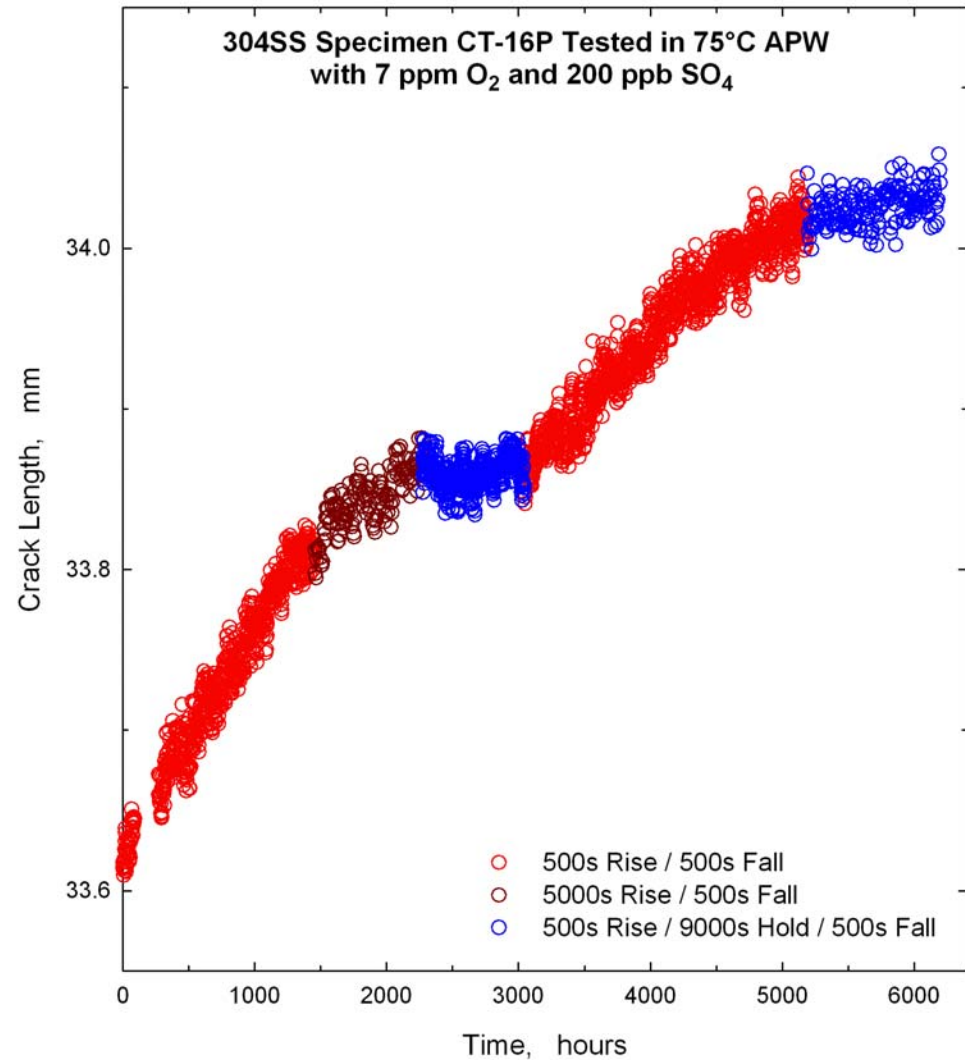
Continuous cycling 500s or 5000s rise
500s rise / 9000s hold / 500s fall



Fully Sensitized 304 SS
75°C APW with 200 ppb SO₄

Continuous cycling
500s rise / 500s fall
5000s rise / 500s fall

500s rise / 9000s hold / 500s fall





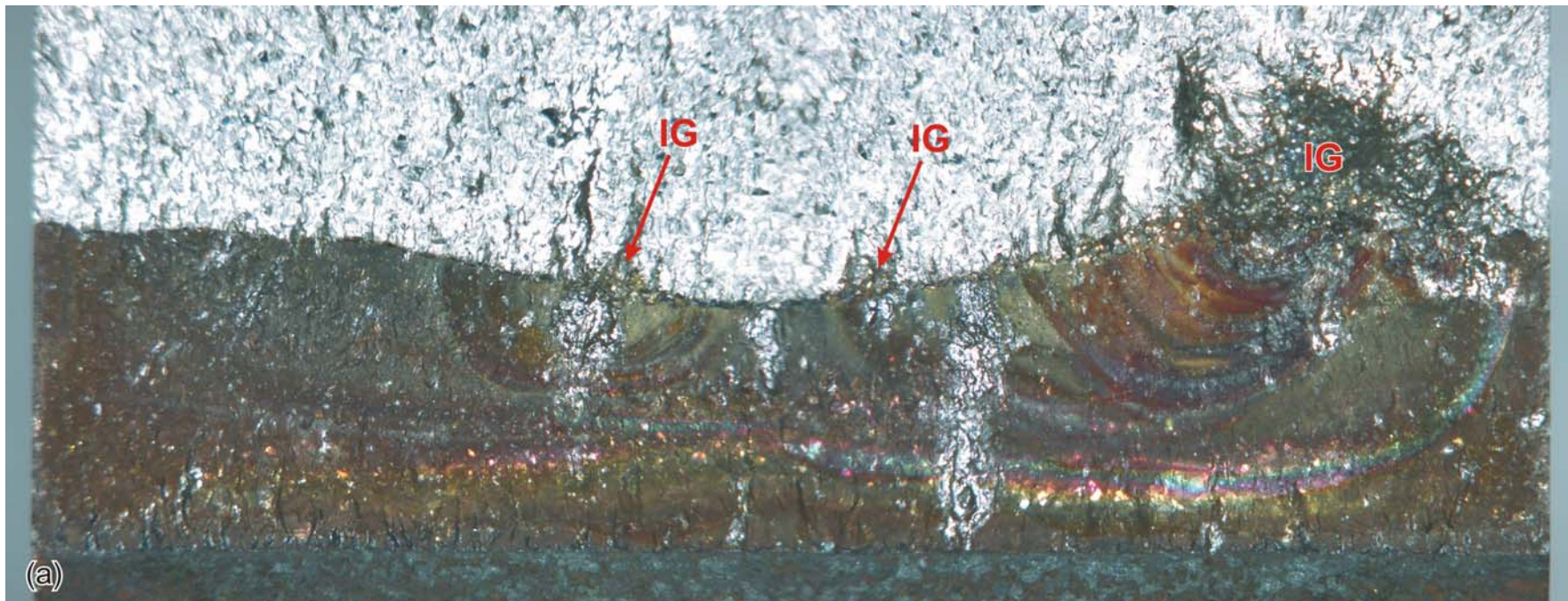
Fully Sensitized 304 SS (CT-16P) Tested in 75°C APW (7 ppm O₂) with 20 ppb SO₄

Continuous cycling produced both TG cracking (corrosion fatigue) and IGSCC.

Average TG crack depth = 0.06 mm.

Average IG crack depth = 0.8 mm (2.5 mm max)

Note discolored rings in vicinity of IG cracks.

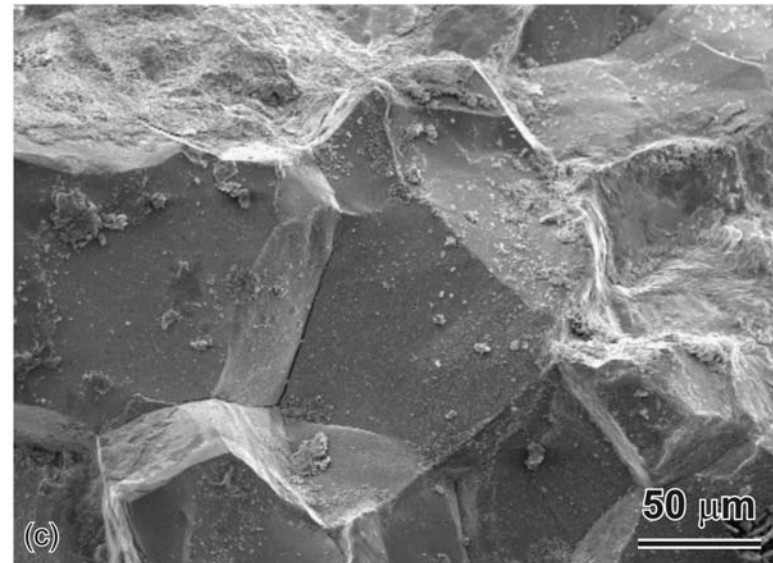
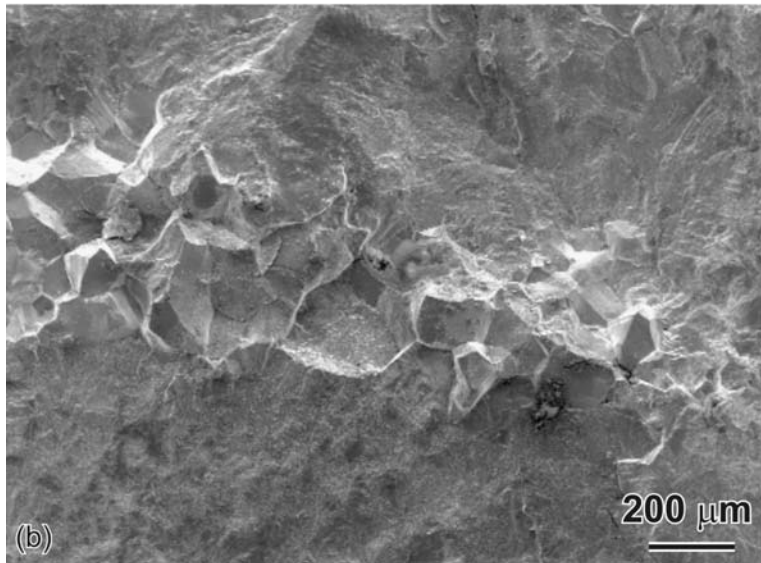
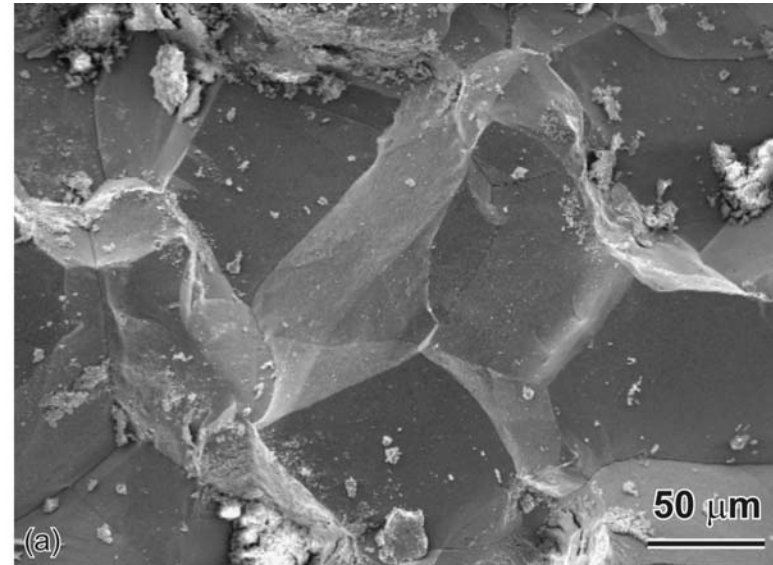




Fully Sensitized 304 SS (CT-16P)
75°C APW: 7 ppm O₂ & 200 ppb SO₄

IGSCC in large IG region (a) &
in narrow IG band (b,c).

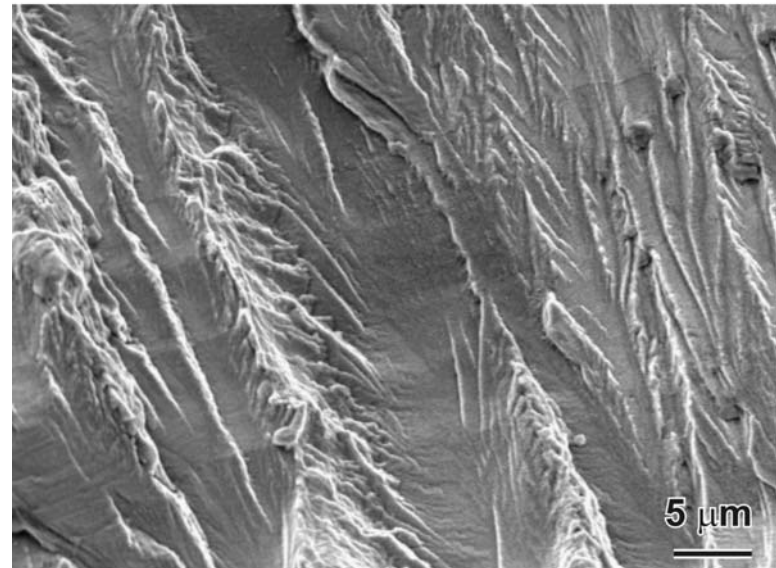
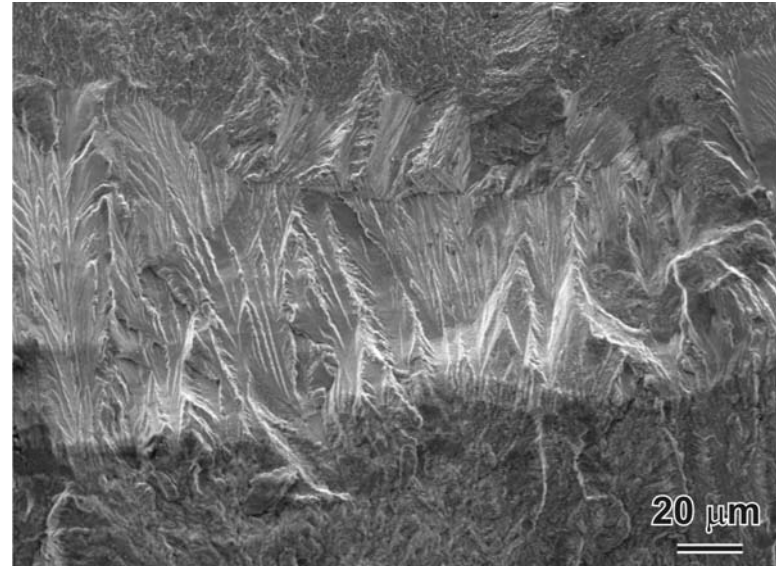
Substantial corrosion debris on IG
fracture surface.





Fully Sensitized 304 SS (CT-16P)
75°C APW: 7 ppm O₂ & 200 ppb SO₄

Narrow band of crystallographic facets
associated with corrosion fatigue
mechanism.





Heavily & Fully Sensitized 304 SS - Tested in
250°C APW with 1 ppm O₂ & 200 ppb SO₄
250°C DPW with 30 cc H₂/kg H₂O & 200 ppb SO₄

Continuous cycling 500s rise
500s rise / 9000s hold / 500s fall



Fully Sensitized 304 SS (WLP2-10)

Narrow IGSCC band during Phase 3 testing in 250°C APW with & without SO₄.

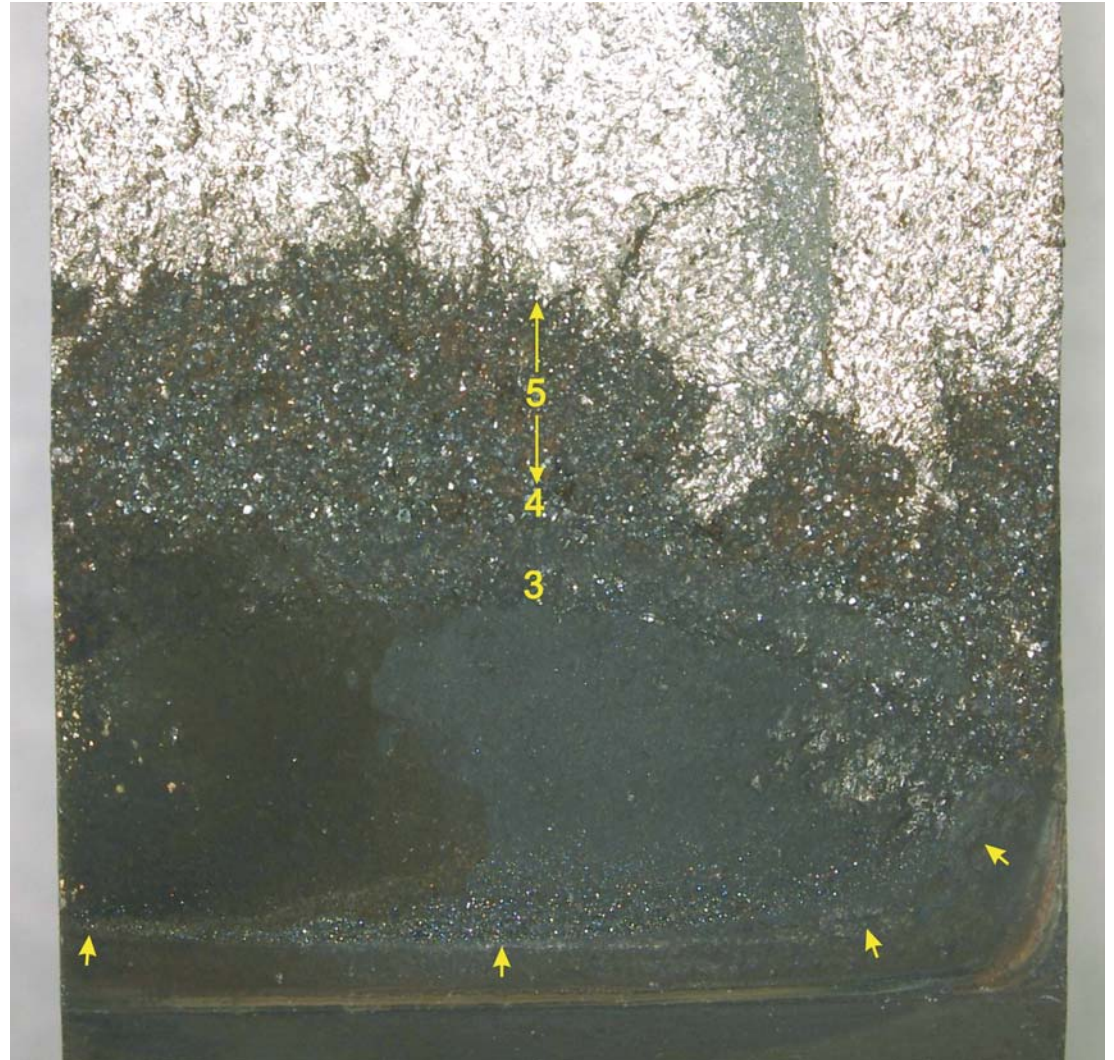
#3 - 500s rise / 500s fall.

Extensive IGSCC during Phase 4/5 testing 250°C APW with SO₄.

#4 - 500s rise / 500s fall.

#5 - 500s rise / 9000s hold / 500s fall.

Fracture surface was covered with a dark oxide and orange-red deposits.





Heavily Sensitized 304 SS (WLP2-6)

Narrow IGSCC band during Phase 3 testing in 250°C APW with & w/o SO_4 .

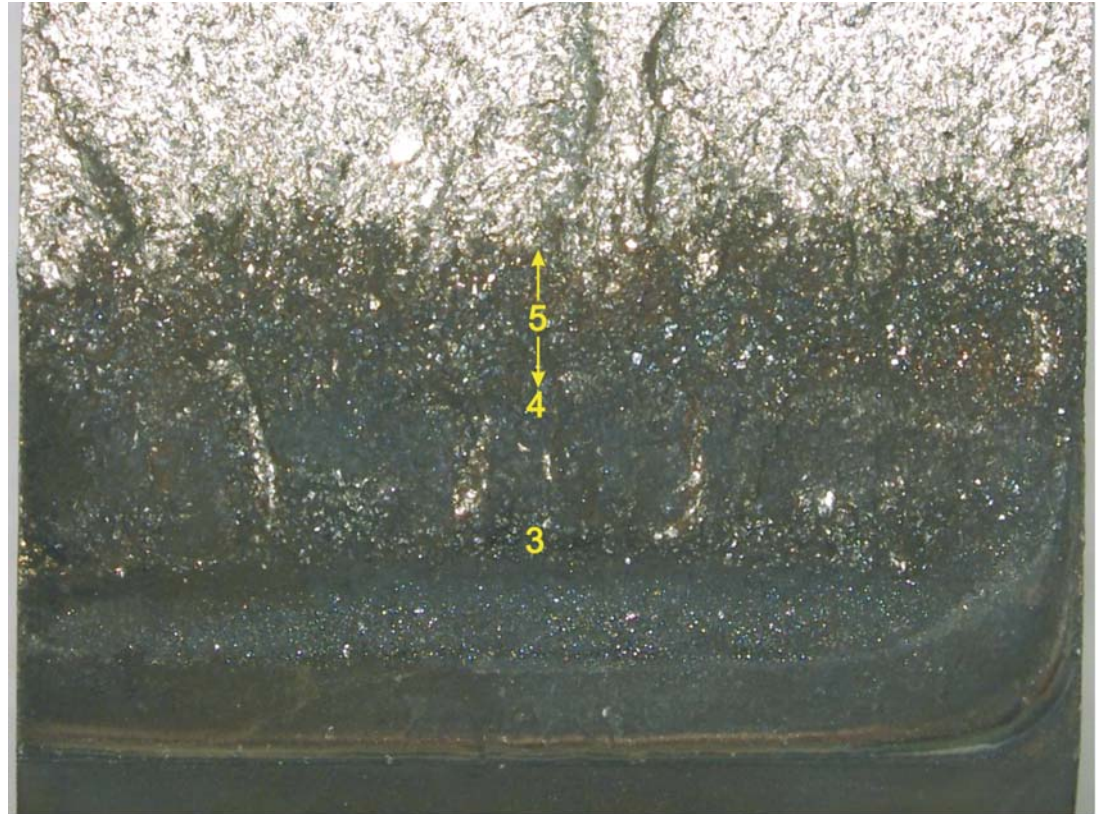
#3 - 500s rise / 500s fall.

Extensive IGSCC during Phase 4/5 testing 250°C APW with SO_4 .

#4 - 500s rise / 500s fall

#5 - 500s rise / 9000s hold / 500s fall.

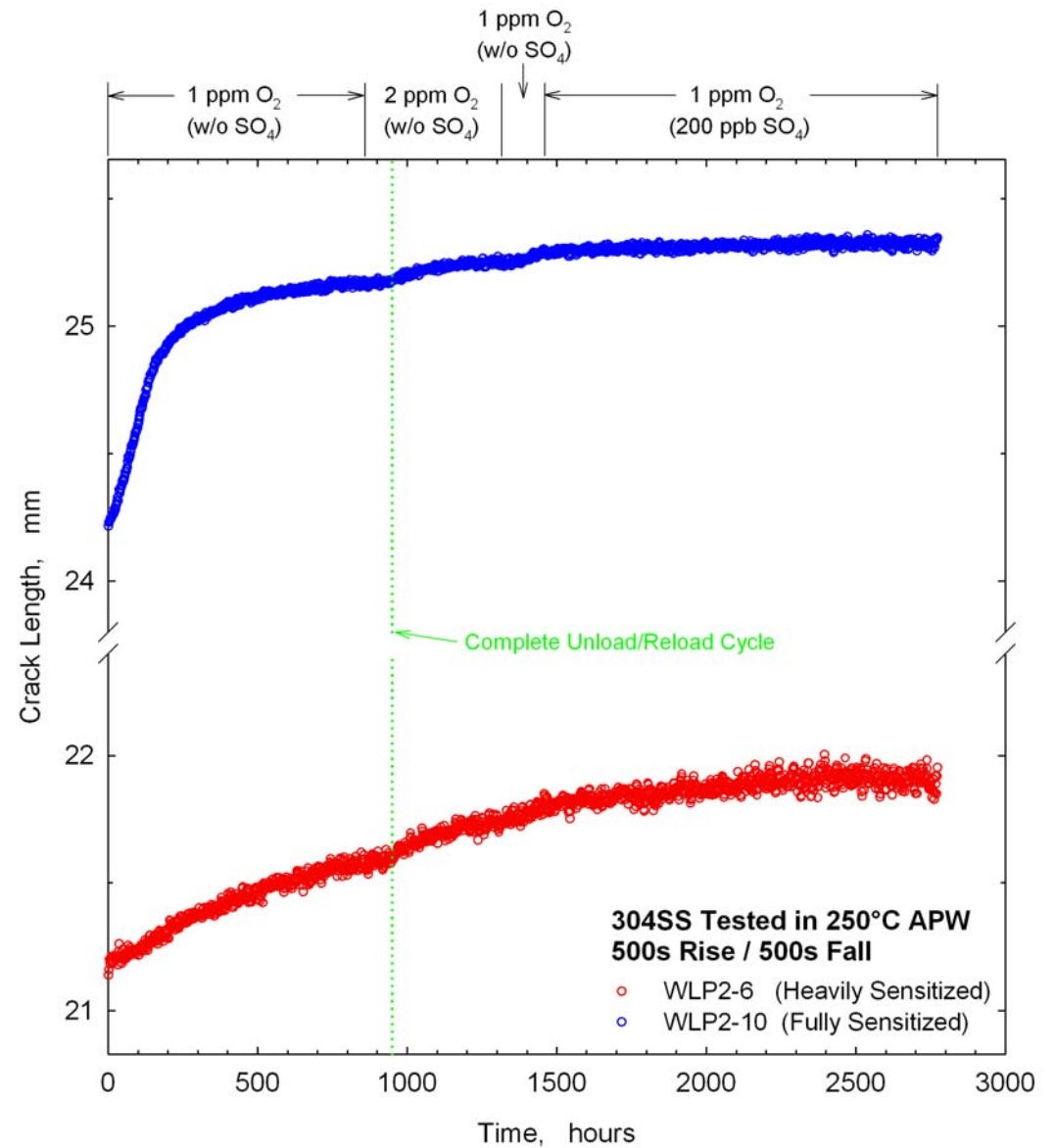
Fracture surface was covered with a dark oxide and orange-red deposits.





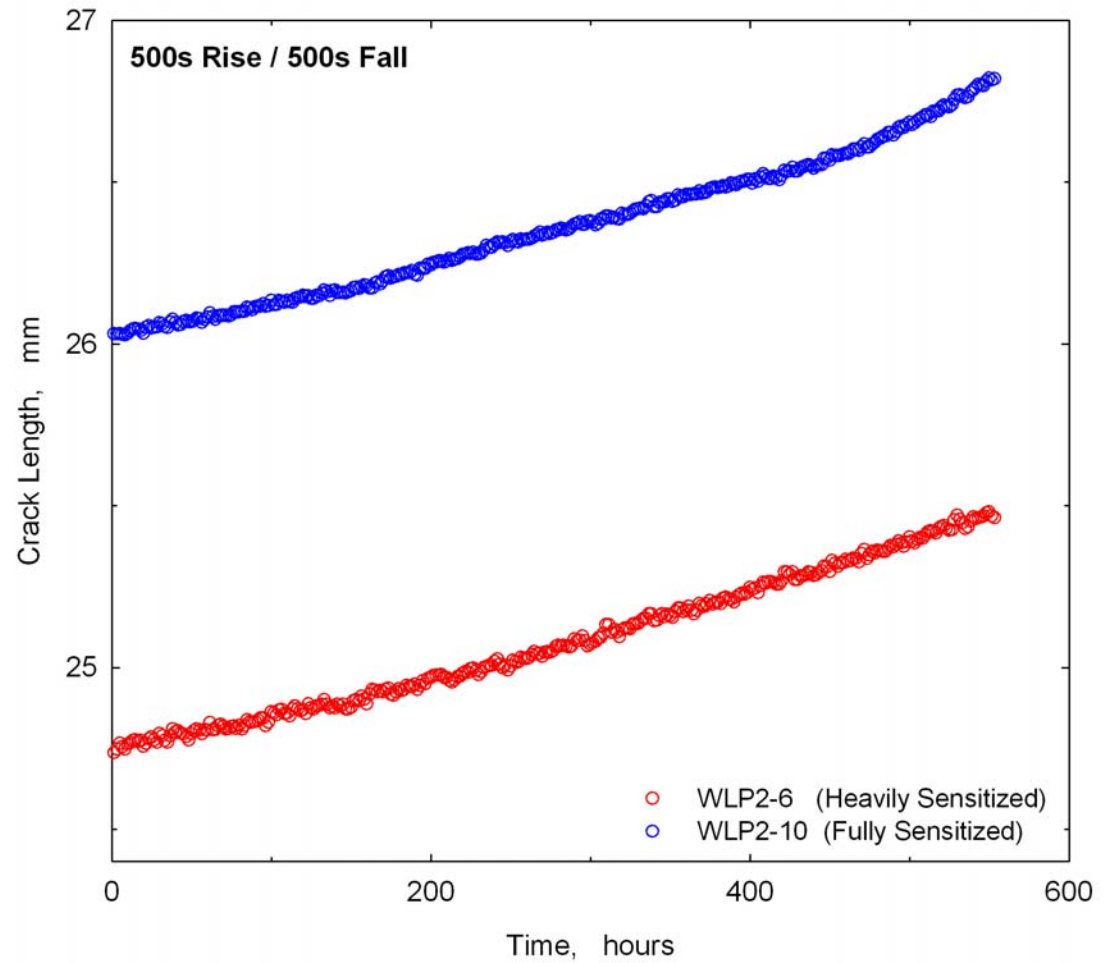
Phase 3 Testing 250°C APW w & w/o SO₄

Continuous cycling
500s rise / 500s fall





Phase 4 Testing
250°C APW with 200 ppb SO₄
Continuous cycling
500s rise / 500s fall

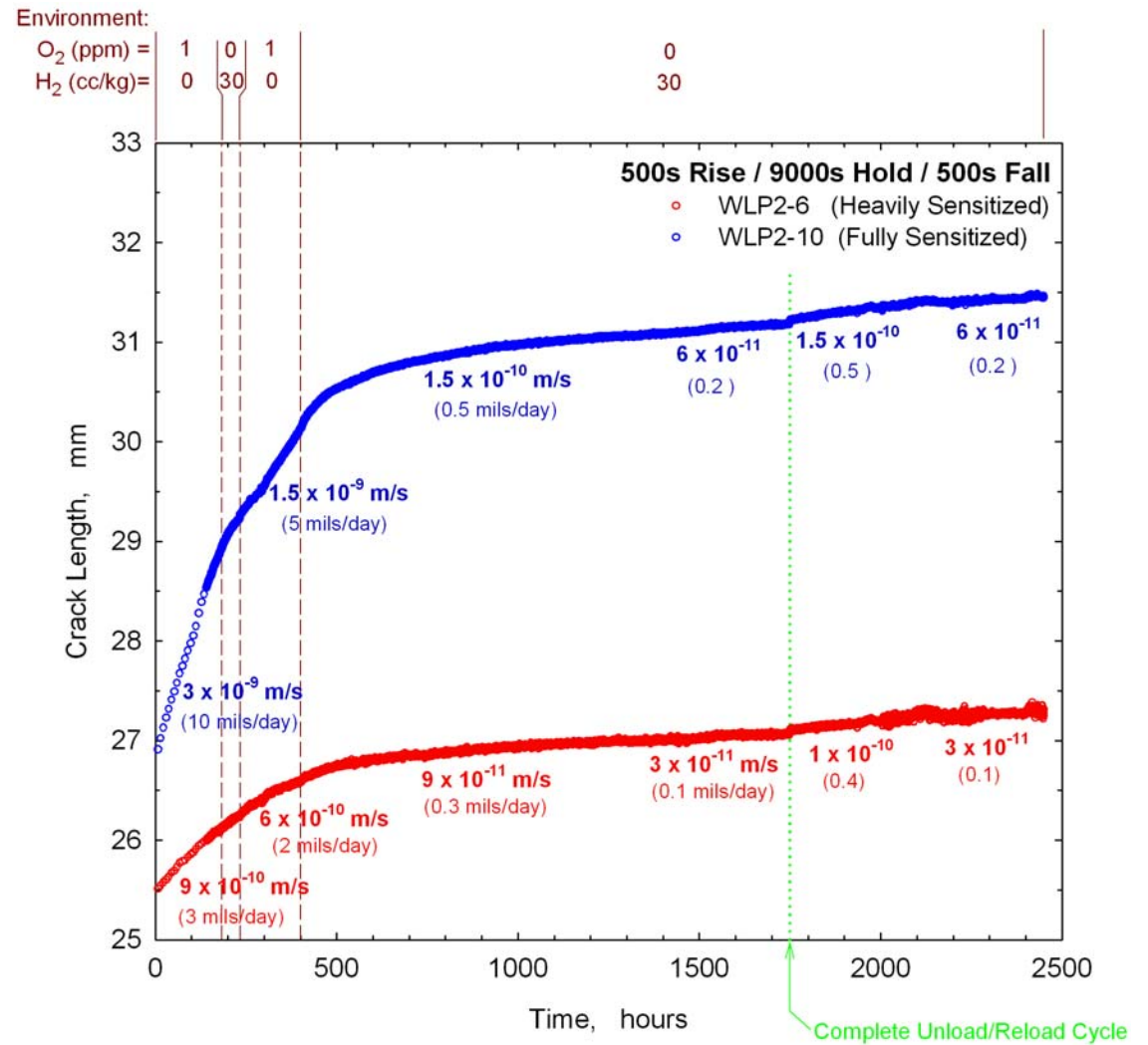




Phase 5 Testing

250°C APW with 200 ppb SO₄ /
250°C DPW with 200 ppb SO₄

500s rise / 9000s hold / 500s fall





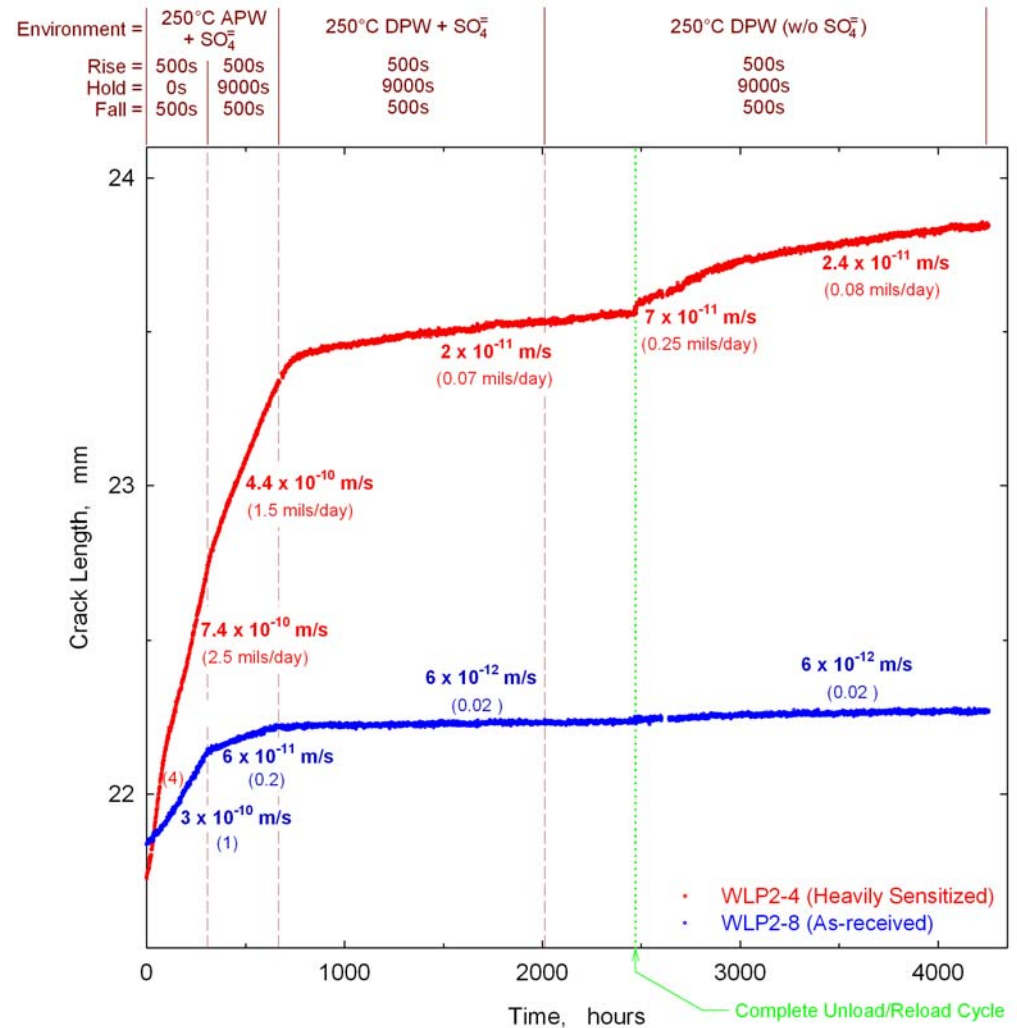
Phase 6a: 250°C APW w 200 ppb SO₄
500s rise / 500s fall

Phase 6b: 250°C APW w 200 ppb SO₄
500s rise / 9000s hold / 500s fall

**Phase 6c: 250°C DPW
w & w/o 200 ppb SO₄**
500s rise / 9000s hold / 500s fall

Cracking mode for Heavily Sensitized
Specimen WLP2-4 tested in 250°C
APW was predominantly **IG**.

Cracking mode for As-Received
Specimen WLP2-8 tested in 250°C
APW was **TG**.





Heavily Sensitized 304 SS (WLP2-6)

Branched IGSCC during Phase 3 testing in 250°C APW with & without SO₄.

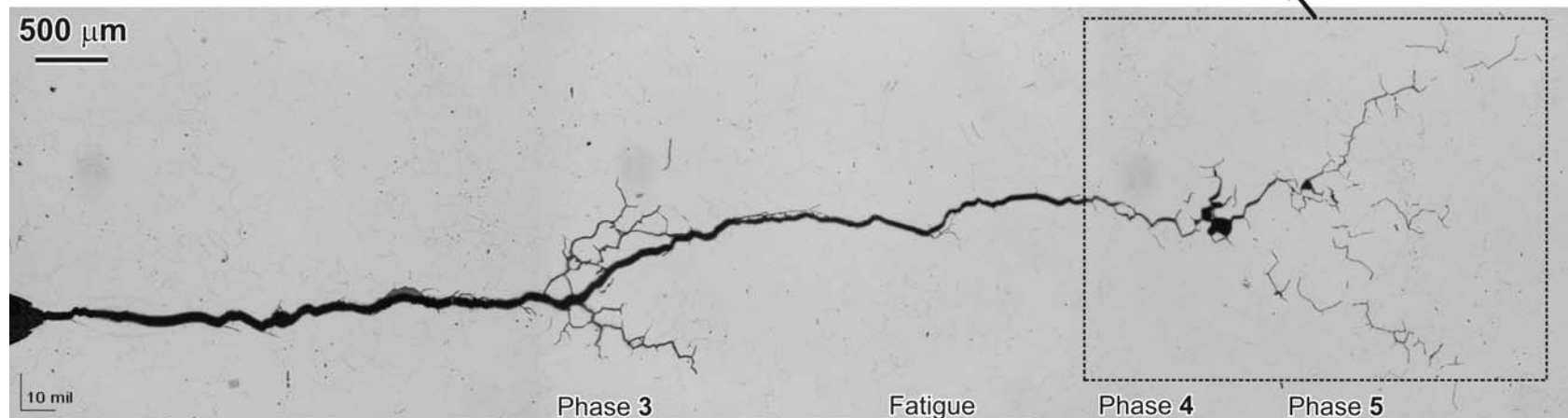
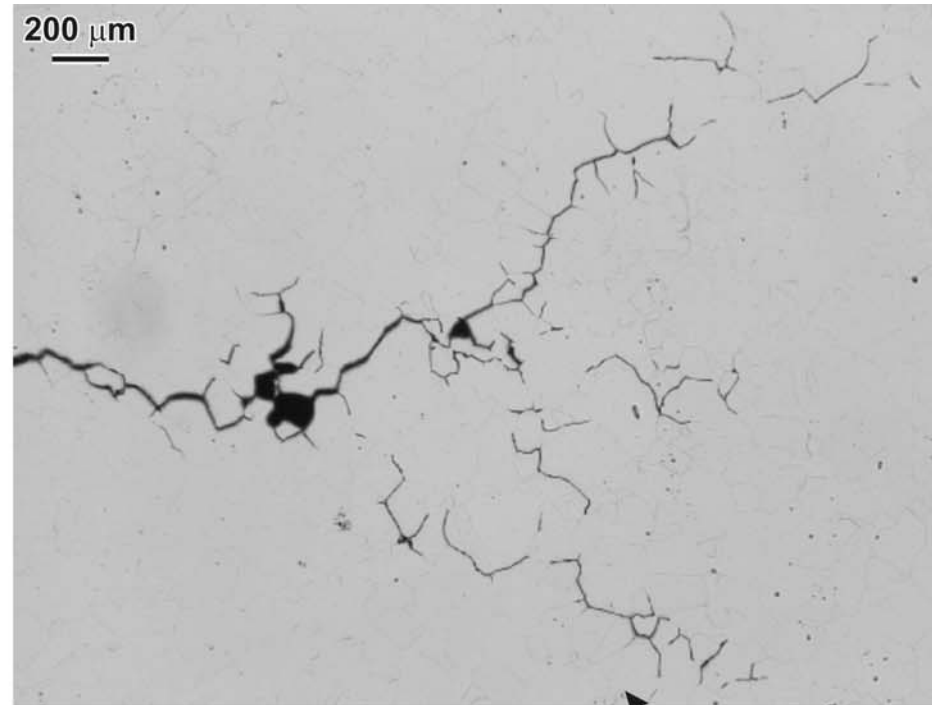
500s rise / 500s fall.

IGSCC during Phase 4 testing in 250°C APW with SO₄.

500s rise / 500s fall.

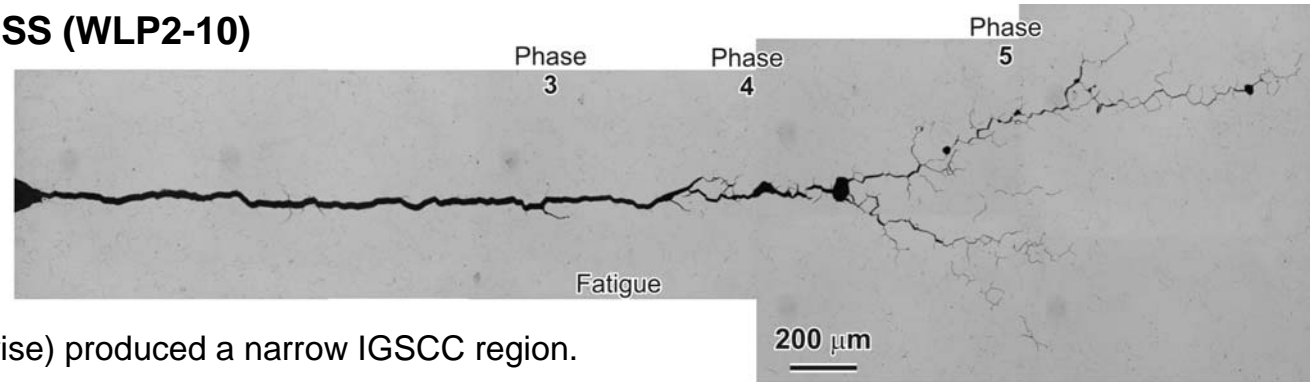
Branched, discontinuous IGSCC during Phase 5 testing in 250°C APW with SO₄.

500s rise / 9000s hold / 500s fall.





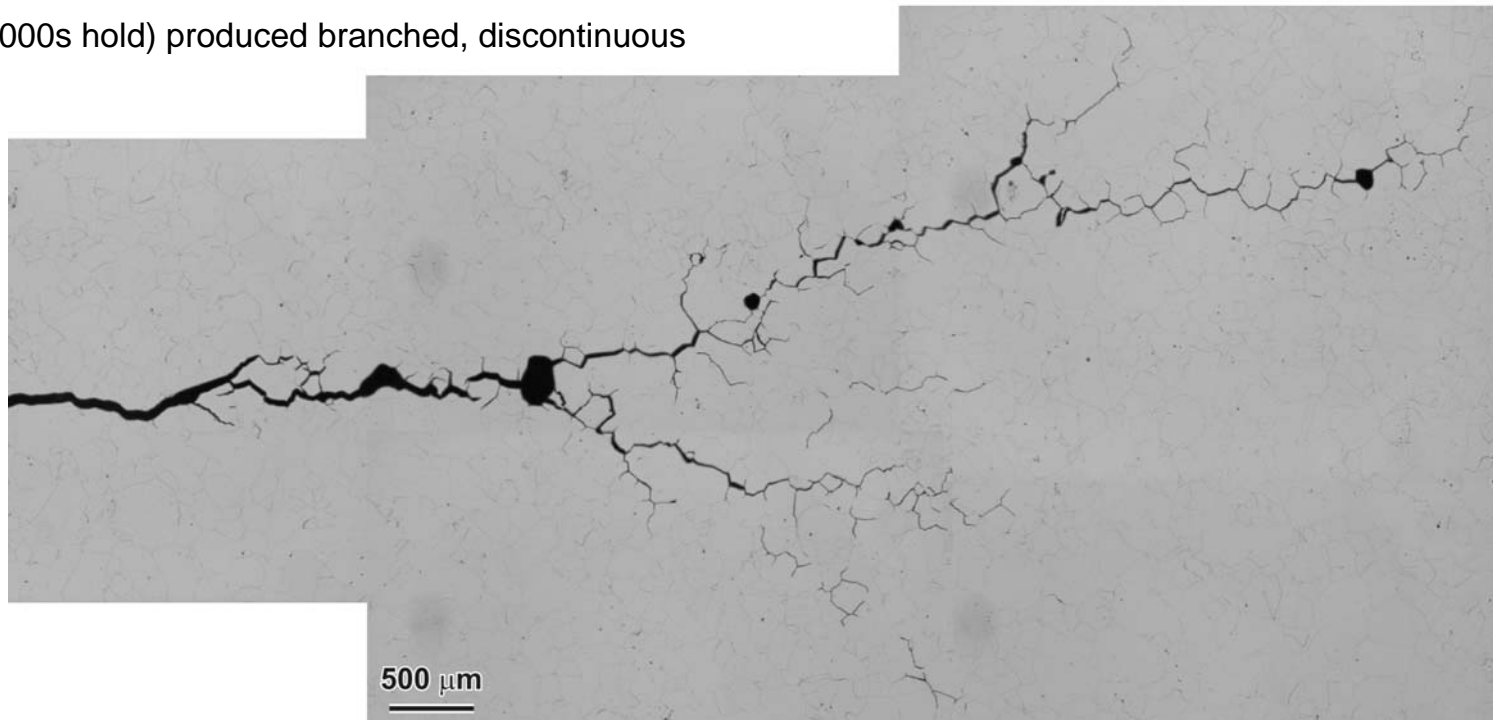
Full Sensitized 304 SS (WLP2-10)



Phase 3 testing (500s rise) produced a narrow IGSCC region.

Phase 4 testing (500s rise) produced IGSCC.

Phase 5 (9000s hold) produced branched, discontinuous IGSCC.

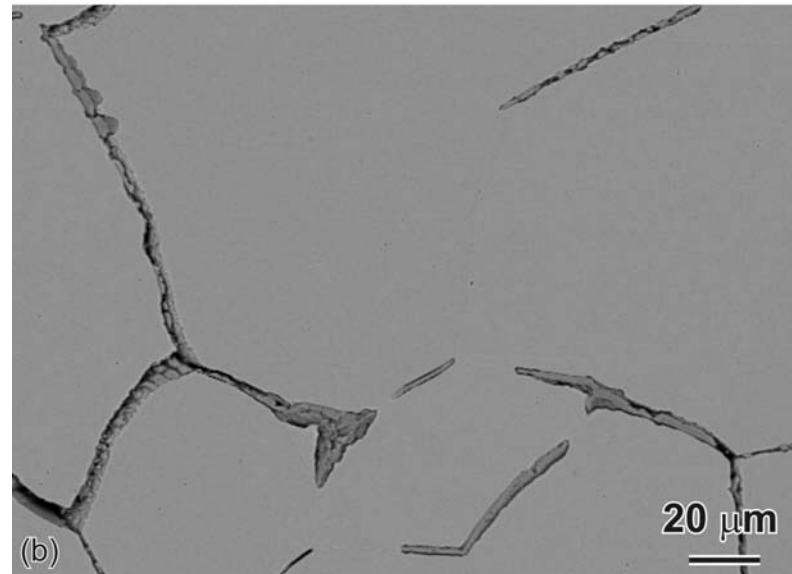
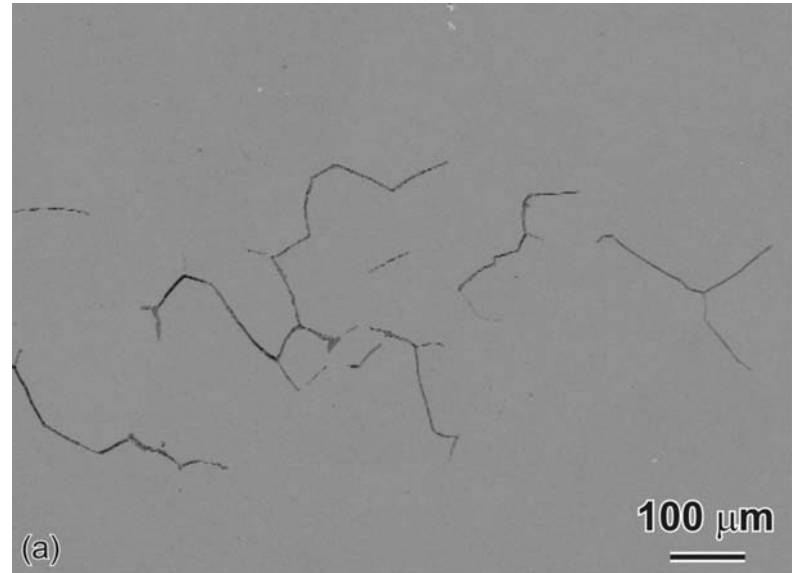




Fully Sensitized 304 SS (WLP2-10)

Far end of IGSCC contained a rather thick oxide,

Suggesting that the IG crack formed in APW.





Heavily Sensitized 304 SS (WLP2-6)

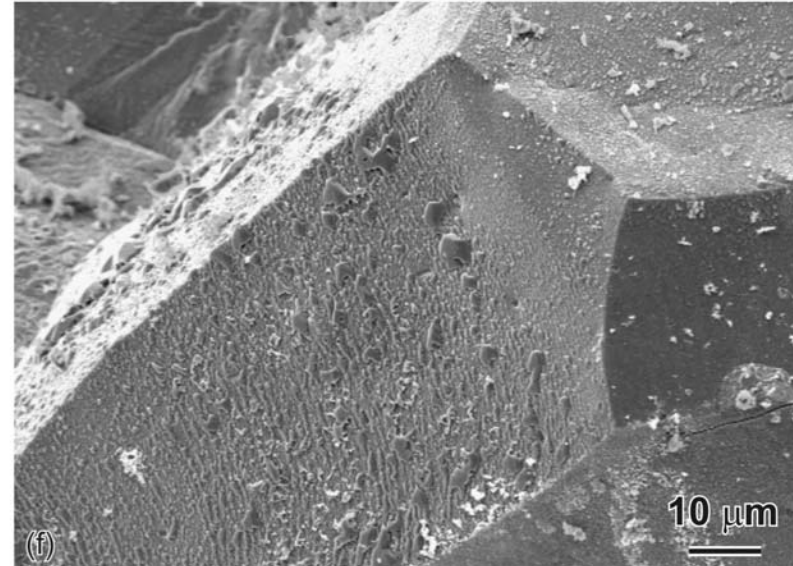
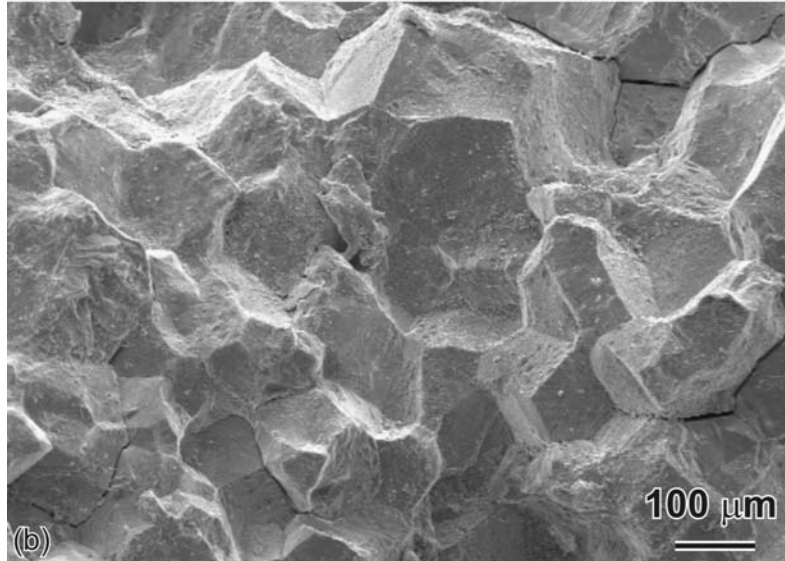
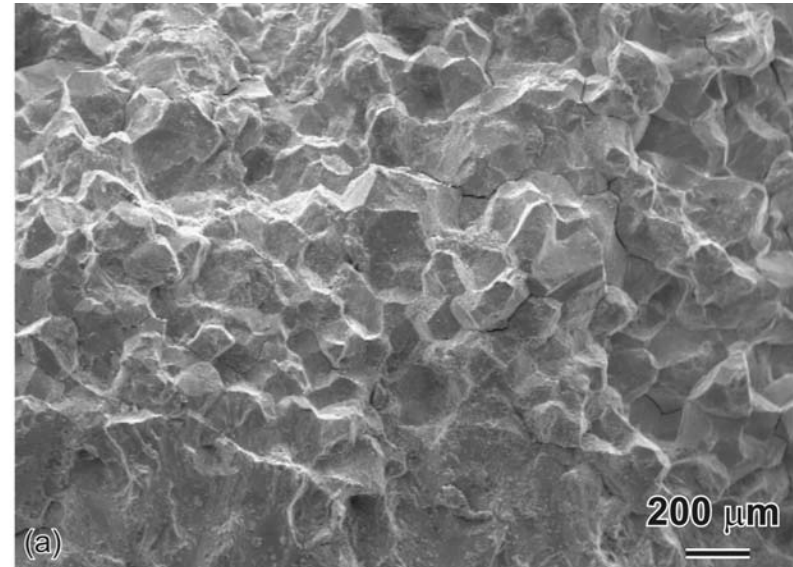
Phase 4

250°C APW with SO_4 .

500s rise / 500s fall.

IGSCC is dominant.

IG faces are covered with a thick oxide film, corrosion debris & large crystals.





Fully Sensitized 304 SS (WLP2-10)

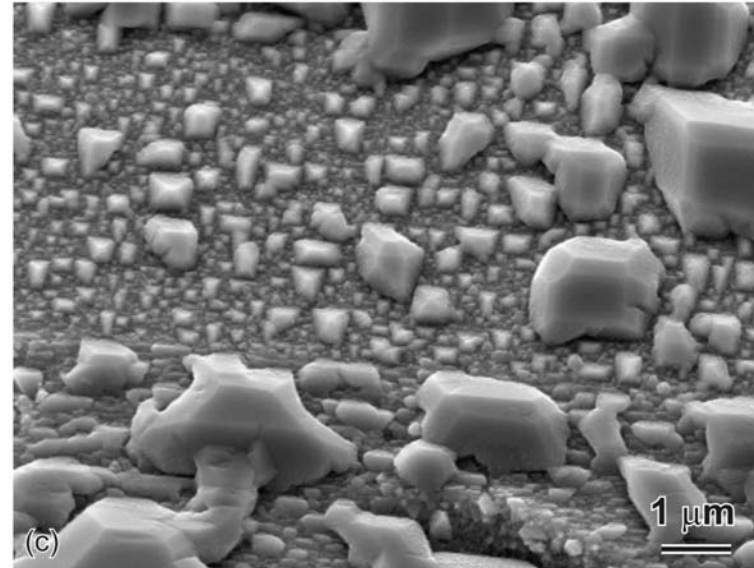
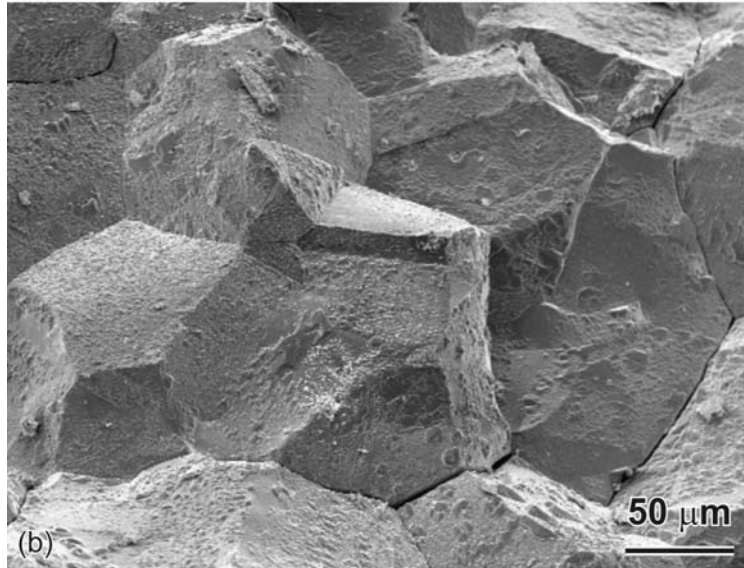
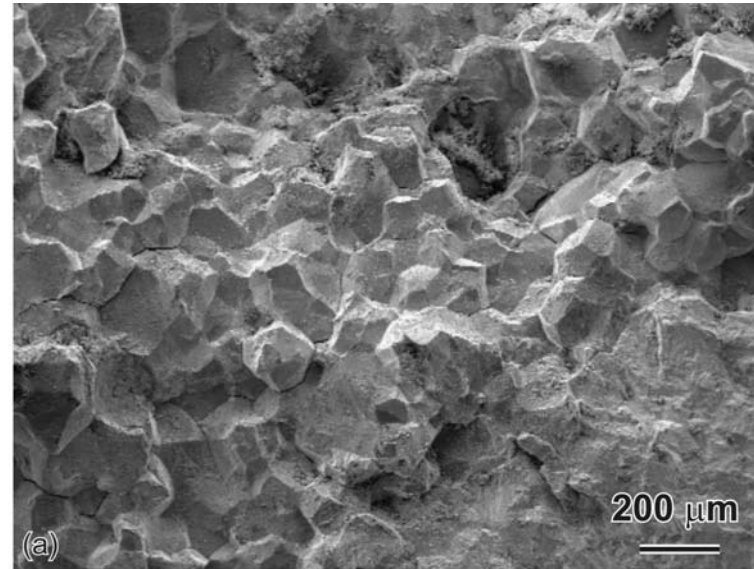
Phase 4

250°C APW with SO_4 .

500s rise / 500s fall.

IGSCC is dominant.

IG faces are covered with a thick oxide film, corrosion debris & large crystals.





**Fully Sensitized 304 SS
Cleaned in ENDOX**

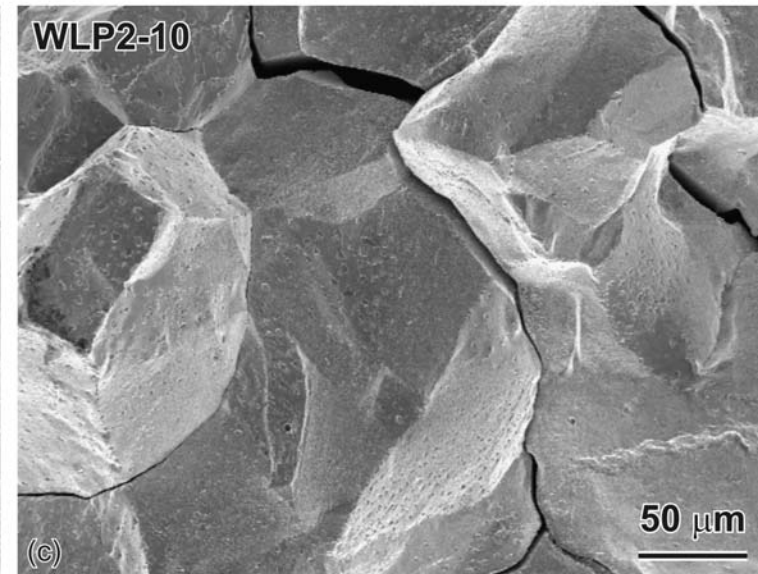
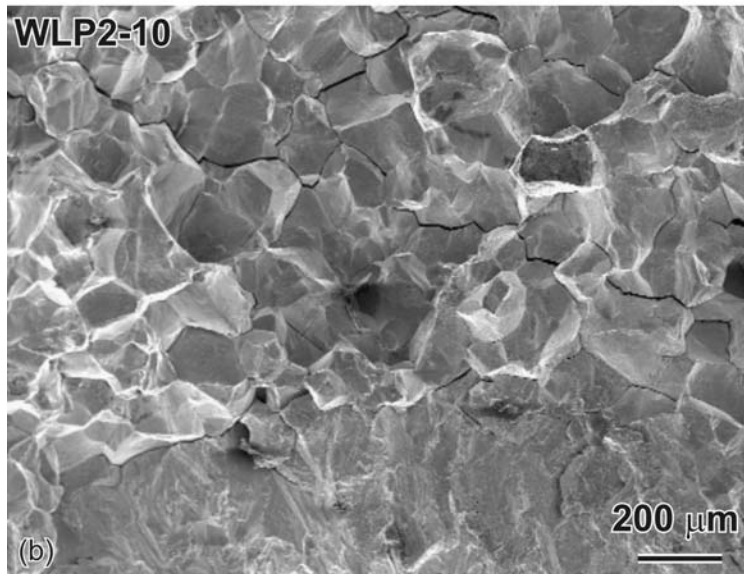
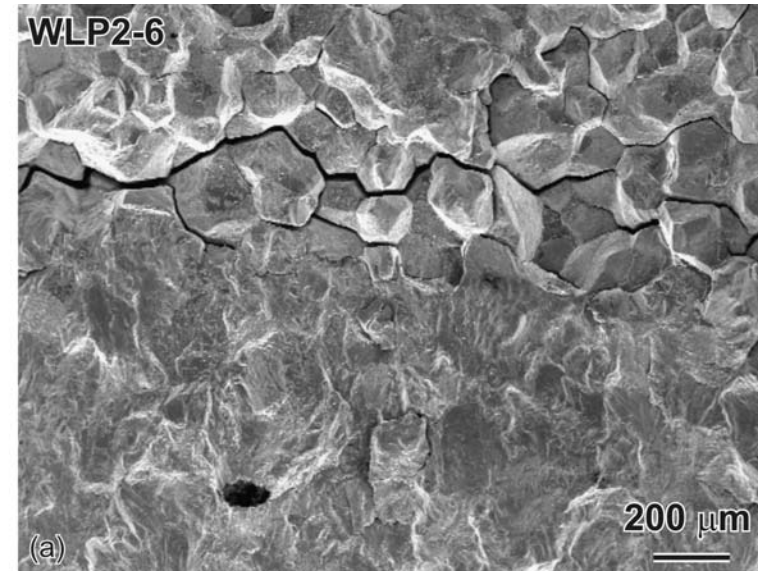
Phase 4

250°C APW with SO₄.

500s rise / 500s fall.

IGSCC is dominant.

Modest secondary IG cracks.





Heavily Sensitized 304 SS (WLP2-6)

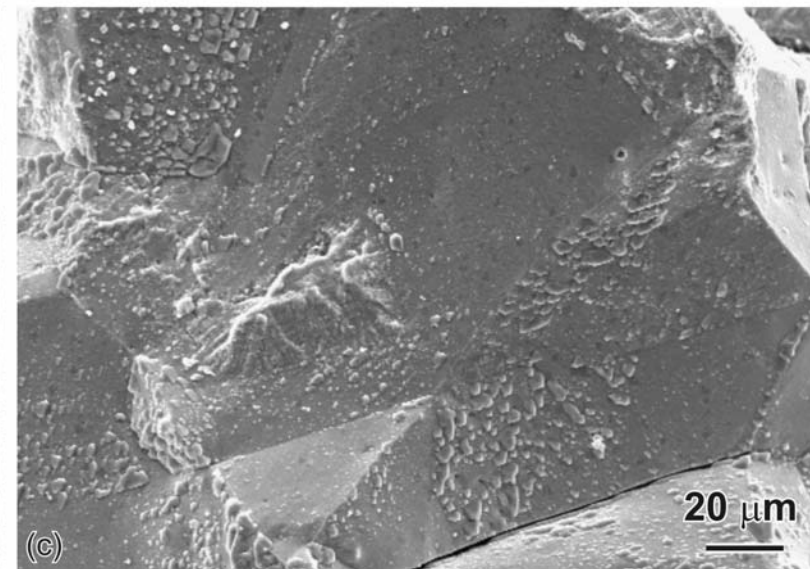
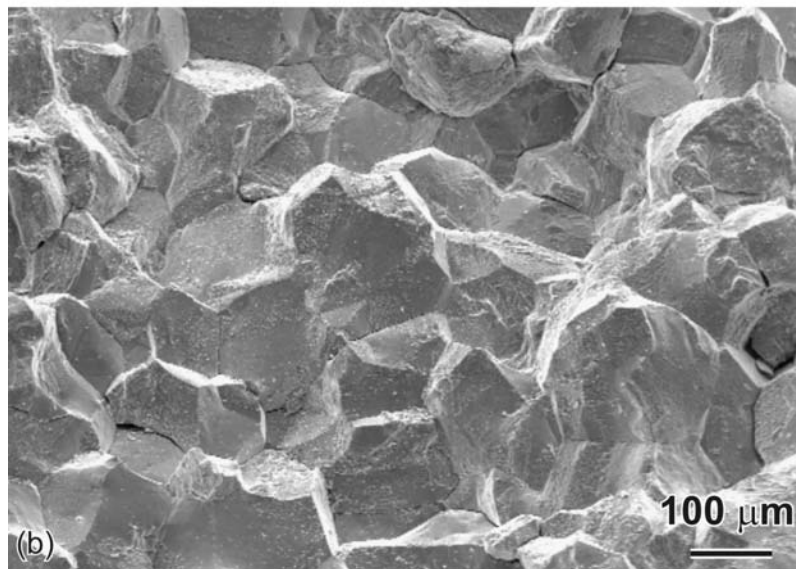
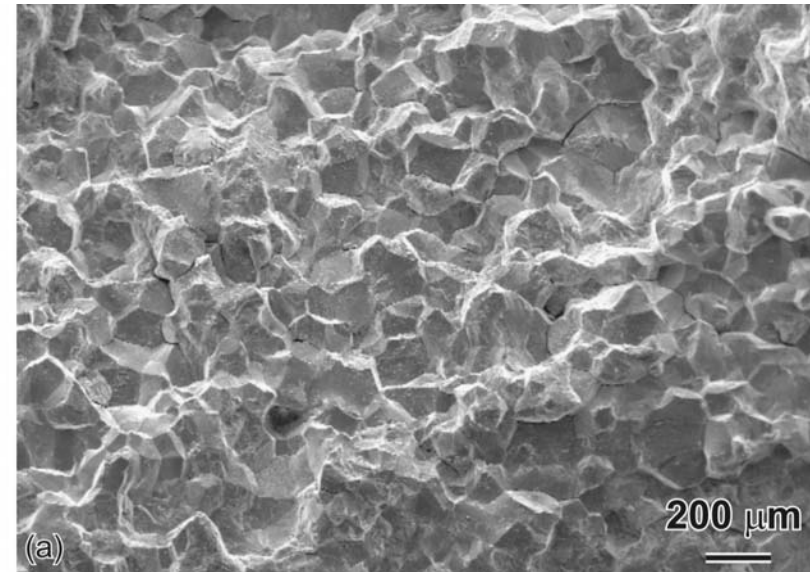
Phase 5

250°C APW with SO_4 .

500s rise / 9000s hold / 500s fall.

IGSCC is dominant.

IG faces are covered with a thick oxide film, corrosion debris & large crystals.





Heavily Sensitized 304 SS (WLP2-6)

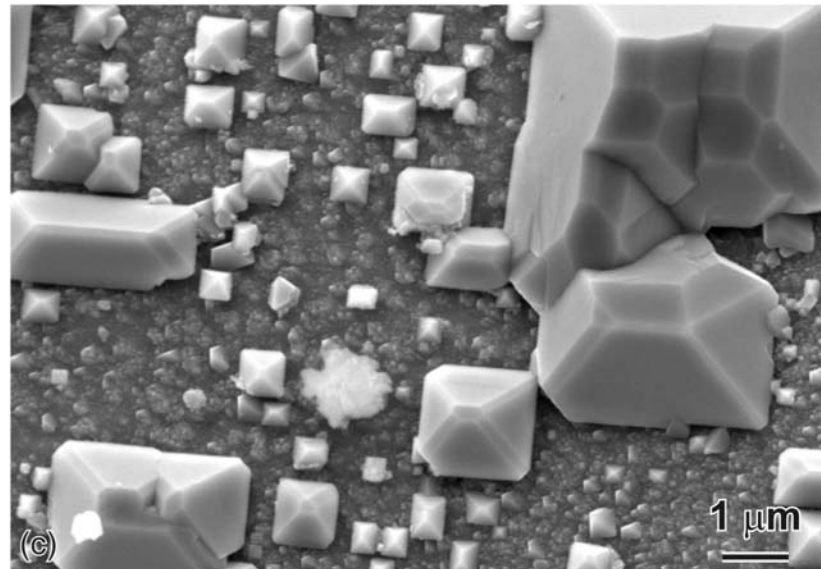
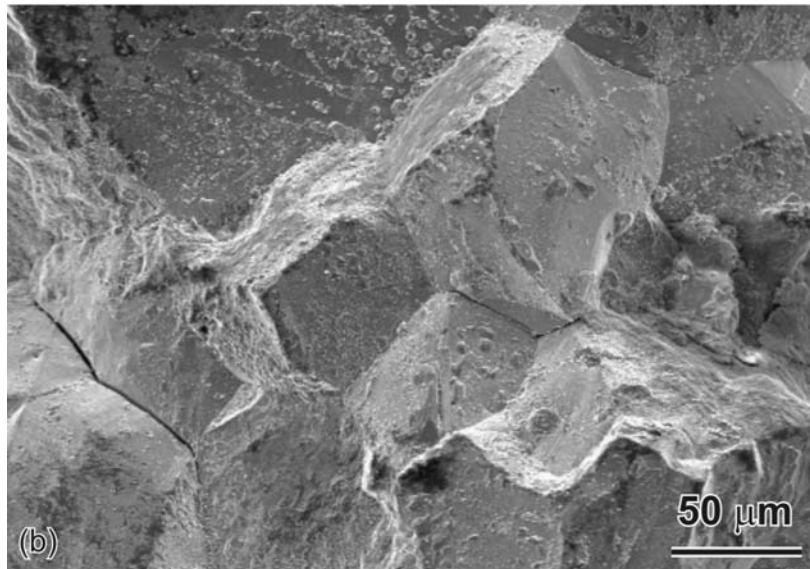
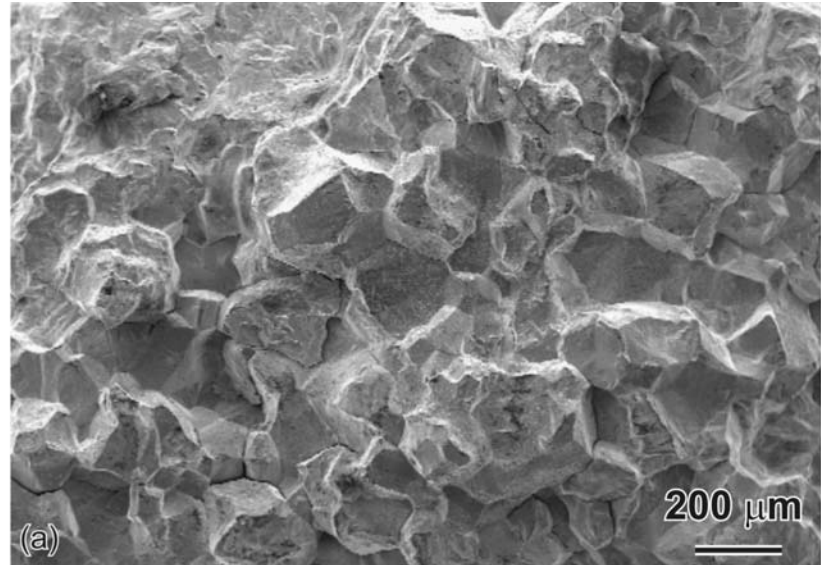
Phase 5

250°C APW with SO_4 .

500s rise / 9000s hold / 500s fall.

IGSCC is dominant.

At far end of IG crack, IG faces are covered with a thick oxide film, corrosion debris & large nickel ferrite crystals.





Fully Sensitized 304 SS (WLP2-10)

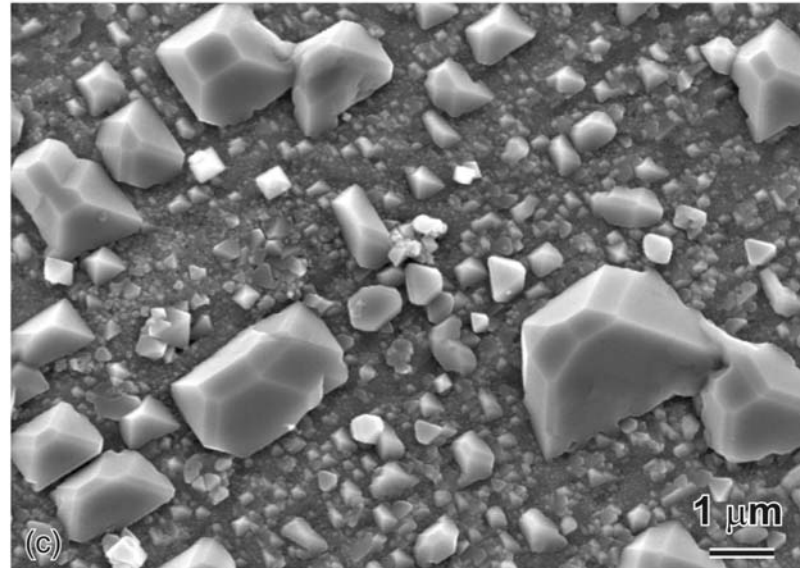
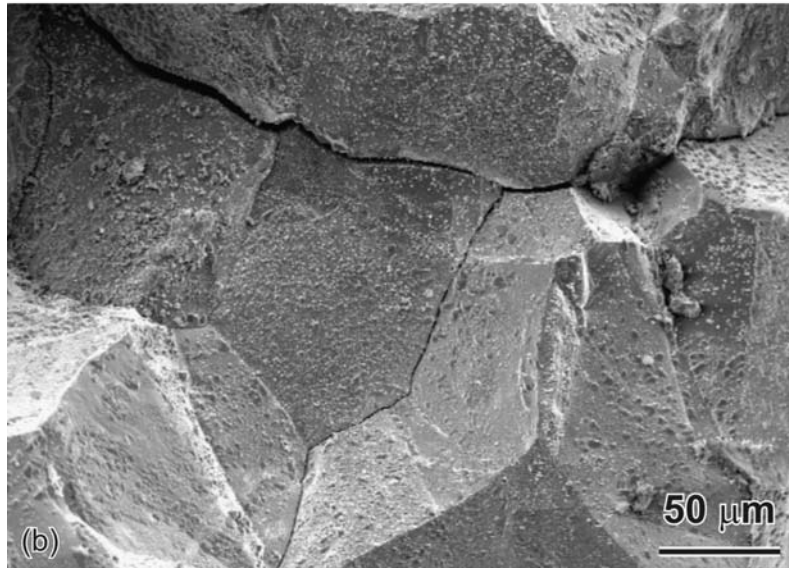
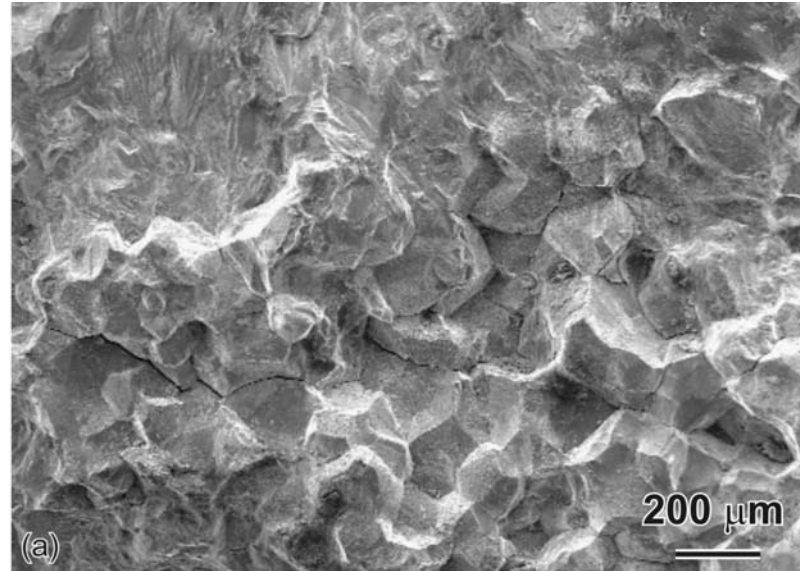
Phase 5

250°C APW with SO_4 .

500s rise / 9000s hold / 500s fall.

IGSCC is dominant.

At far end of IG crack, IG faces are covered with a thick oxide film, corrosion debris & large nickel ferrite crystals.





Heavily Sensitized 304 SS (WLP2-6)

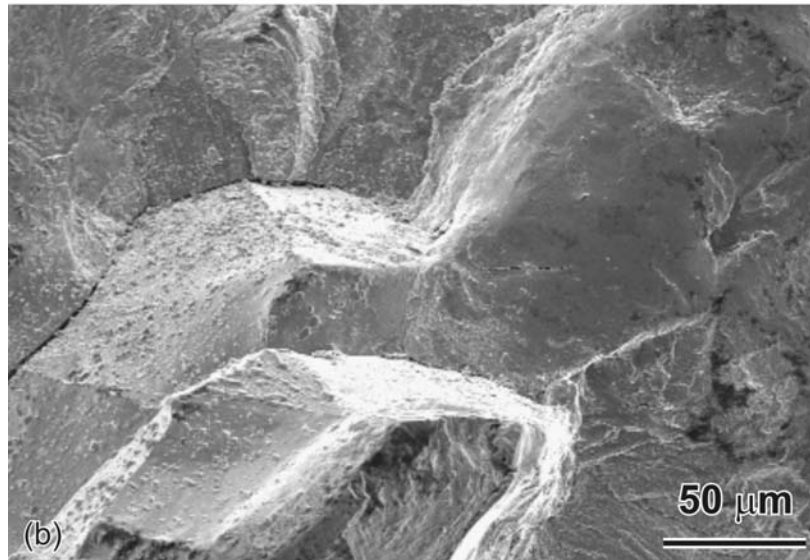
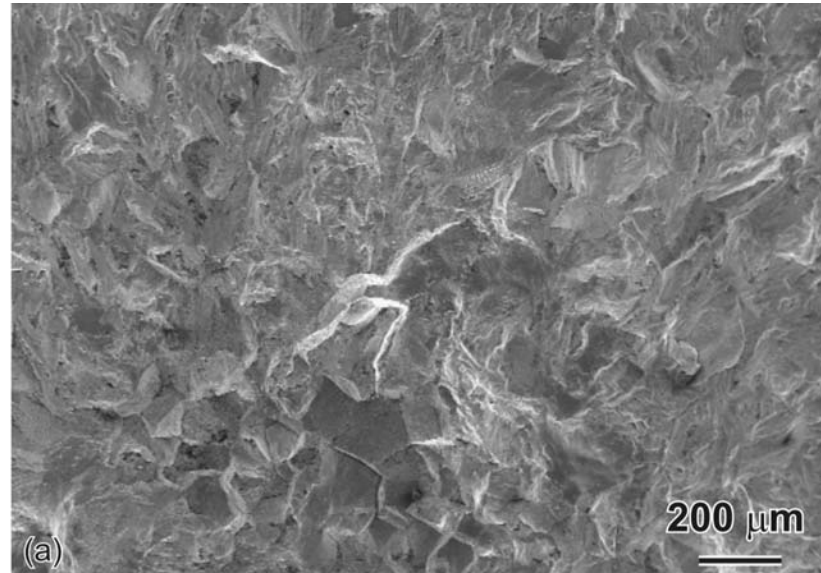
Phase 5

250°C APW / DPW with SO_4 .

500s rise / 9000s hold / 500s fall.

Beyond the IGSCC with large ferrite crystals (APW), the fracture surface exhibits broad TG facets (DPW).

The fracture surface generated in 250°C DPW was predominantly TG, but isolated IG islands were found.





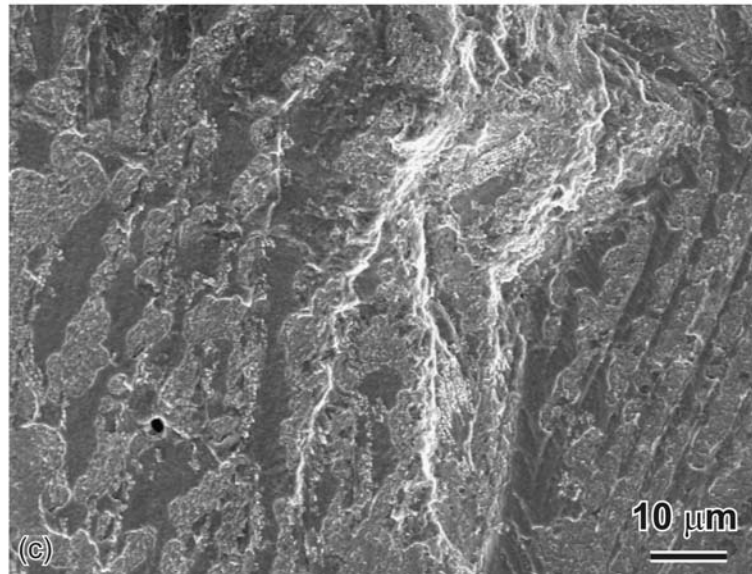
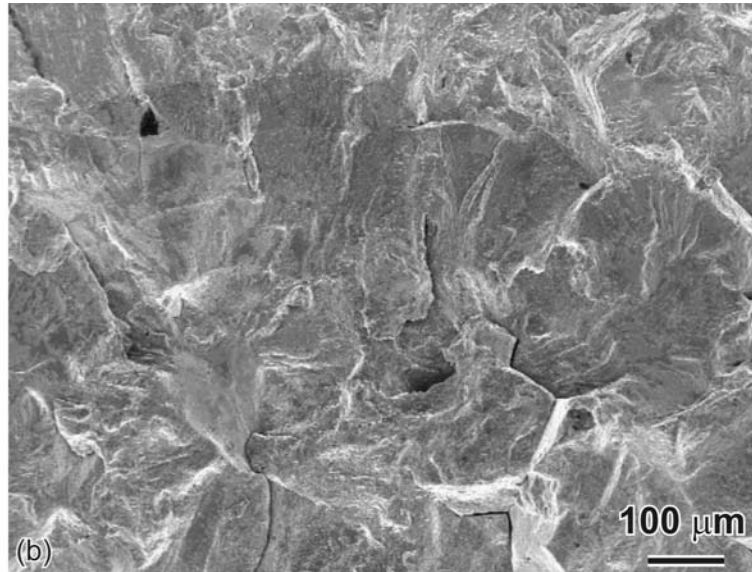
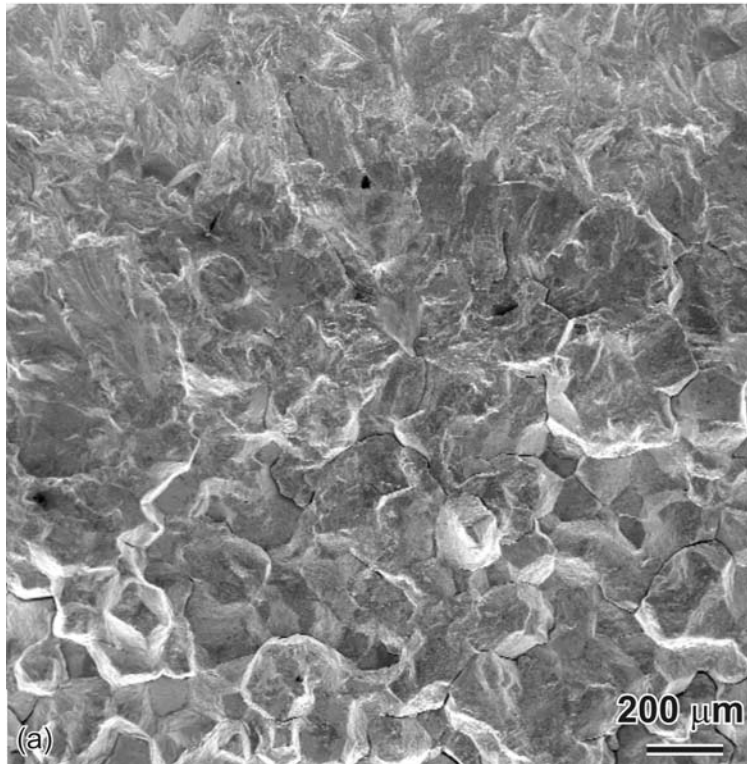
Heavily Sensitized 304 SS (WLP2-6) Cleaned in ENDOX

Phase 5

250°C APW / DPW with SO_4 .

500s rise / 9000s hold / 500s fall.

Broad TG facets just beyond IG crack
were formed in 250°C DPW





20% Cold Rolled 304 SS
Tested in 250°C APW & 250°C DPW

In 250°C APW (0.2 & 1 ppm O₂), IGSCC readily incubated from a TG crack.

Rapid IGSCC continued when the water was deaerated and hydrogenated to 25 cc H₂/kg H₂O.



20% Cold Rolled 304 SS Specimen CT-2CR Tested in 250°C APW / 250°C DPW

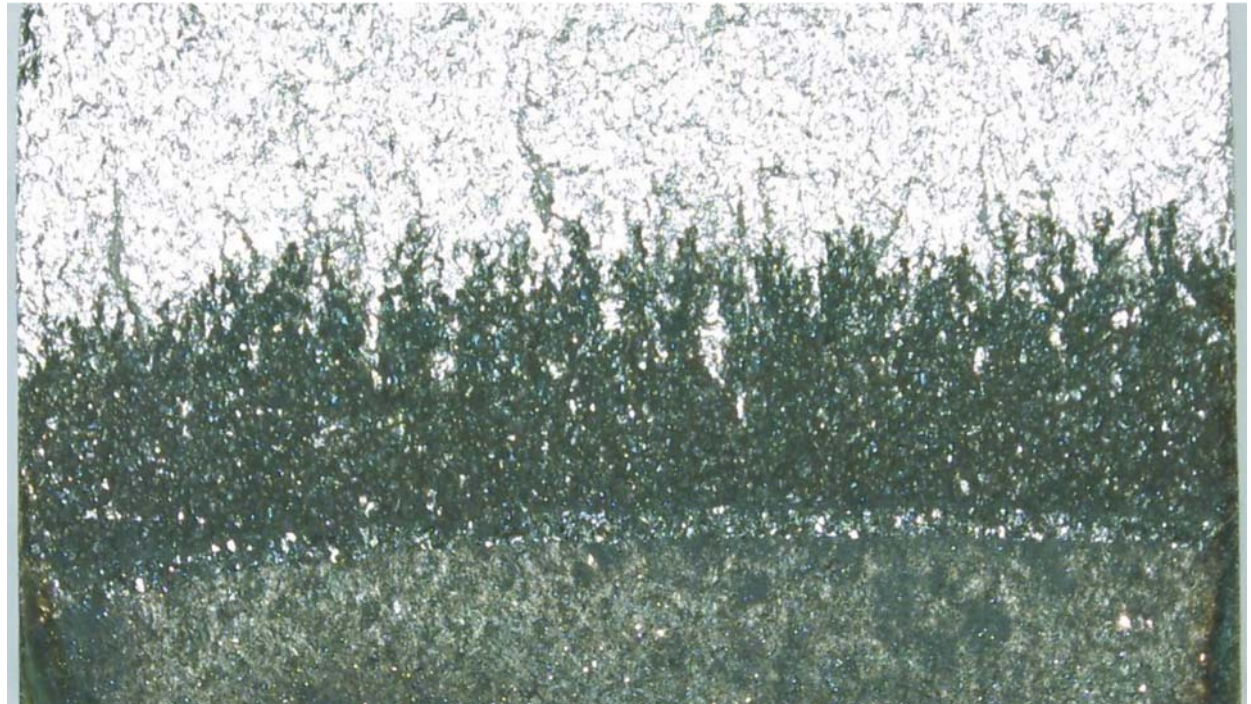
Specimen was tested in
250°C APW / 250°C DPW.

APW - 500s / 5000s rise

APW - 9000s hold

DPW - 9000s hold

Note unbroken ligaments
in IG region, especially in
IG region generated in
DPW.

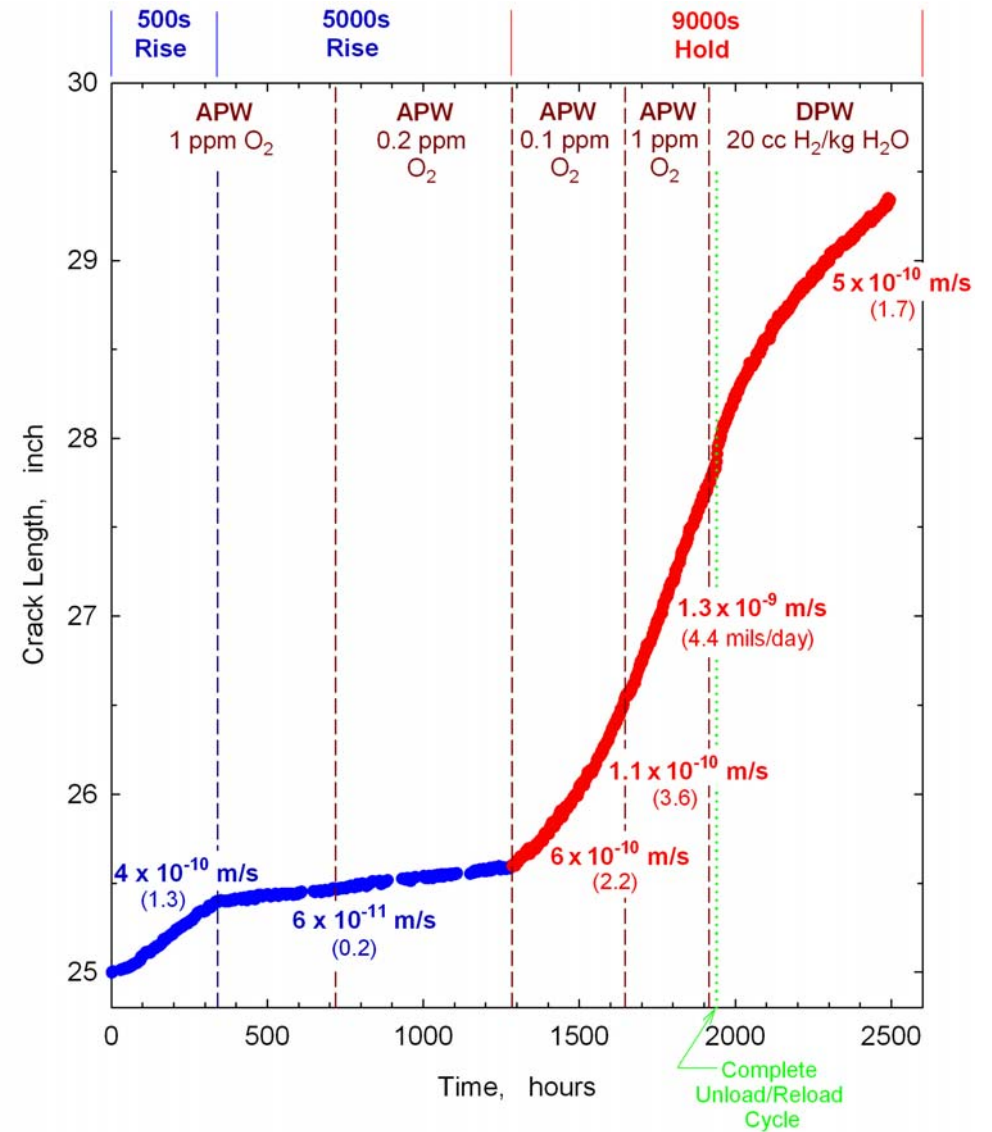


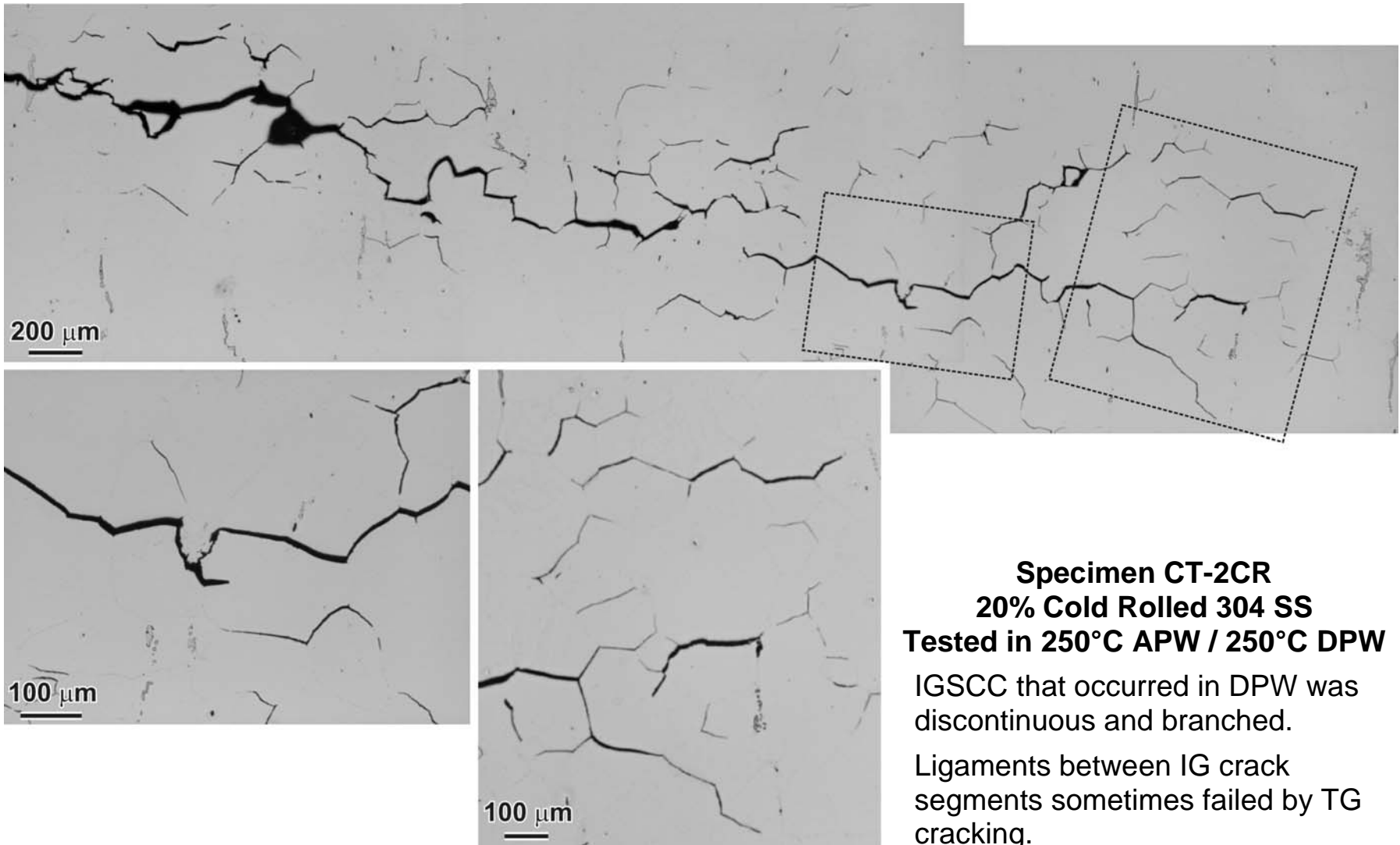


20% Cold Rolled 304 SS (CT-2CR)
Tested in 250°C APW / DPW

Continuous Cycling: 500s-5000s rise
TG cracking mode

500s rise / 9000s hold / 500s fall
IGSCC

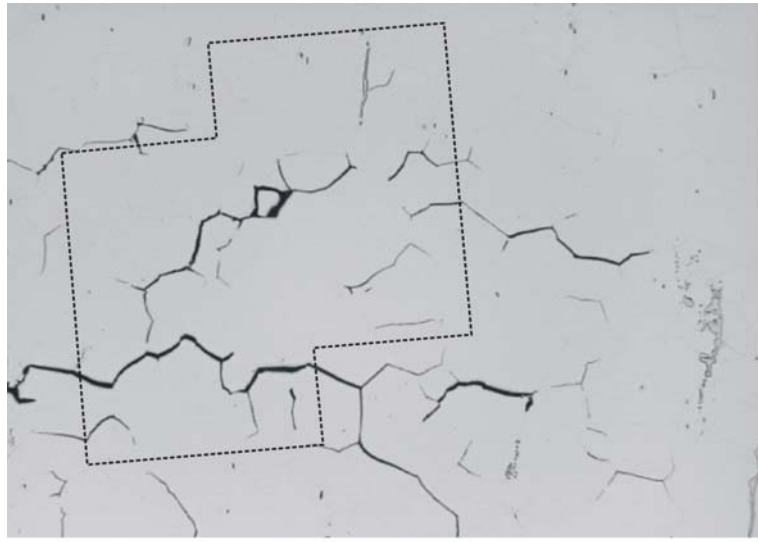




Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW / 250°C DPW

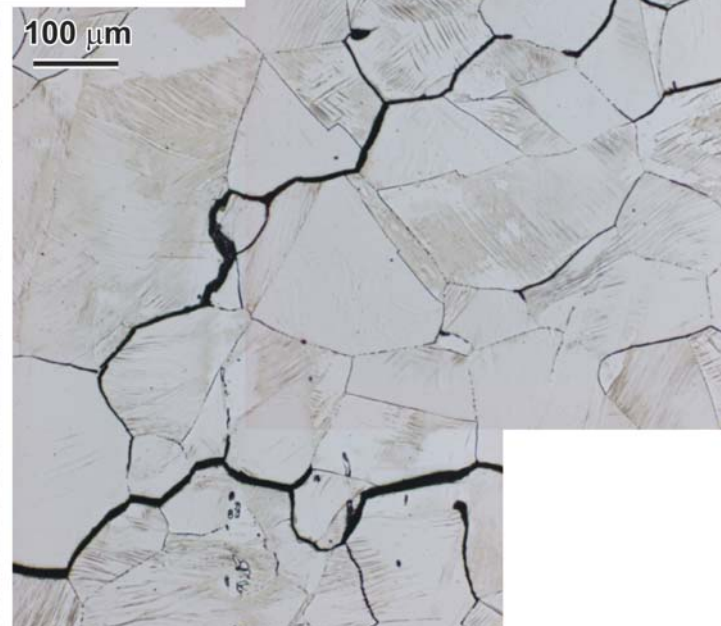
IGSCC that occurred in DPW was discontinuous and branched.

Ligaments between IG crack segments sometimes failed by TG cracking.



Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW / 250°C DPW

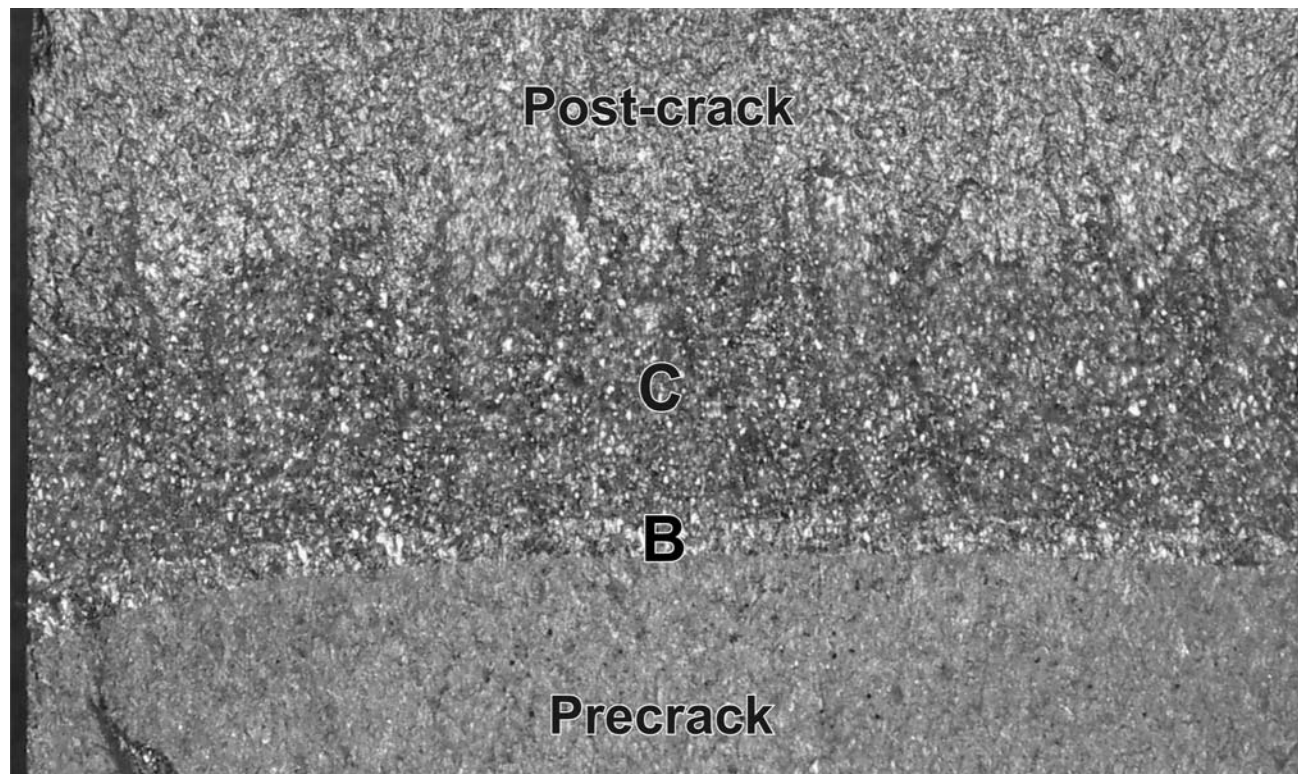
SCC in 250°C DPW produced
discontinuous & branched IGSCC.





20% Cold Rolled 304 SS Specimen CT-2CR
Tested in 250°C APW / 250°C DPW

Fracture surface cleaned in ENDOX solution.

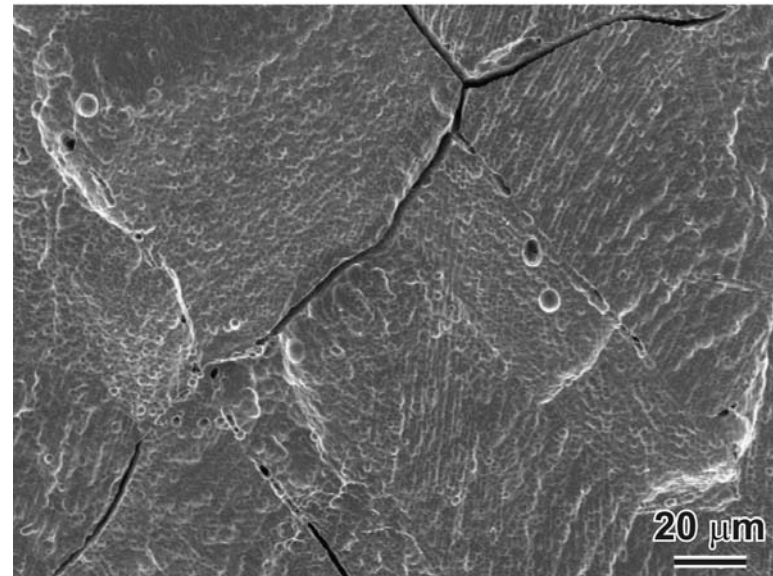
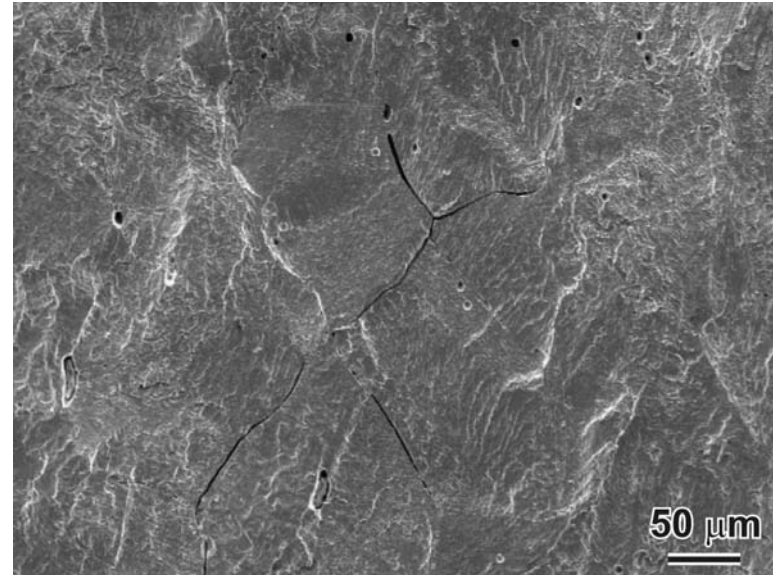




**Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW**

Primary fatigue precrack was TG; however,
there was evidence of secondary IG cracks.

Secondary IG cracks suggest that 20%
CW may have degraded cohesive
strength of GBs.

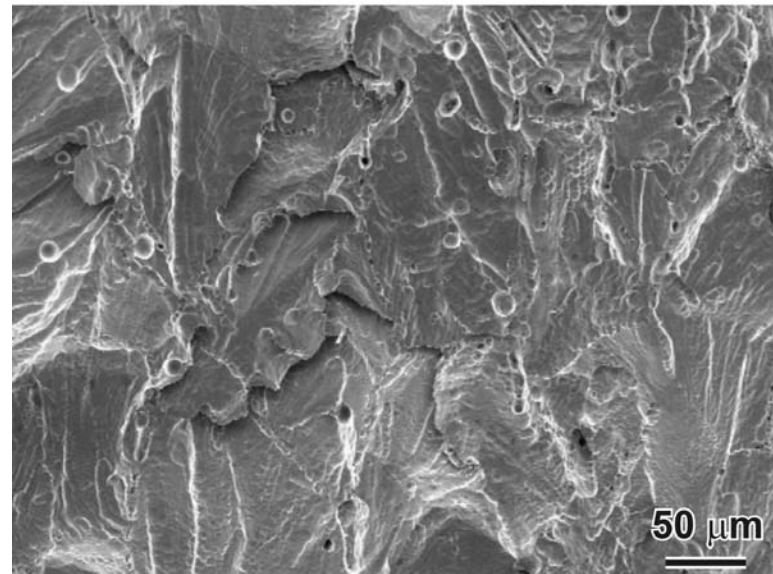
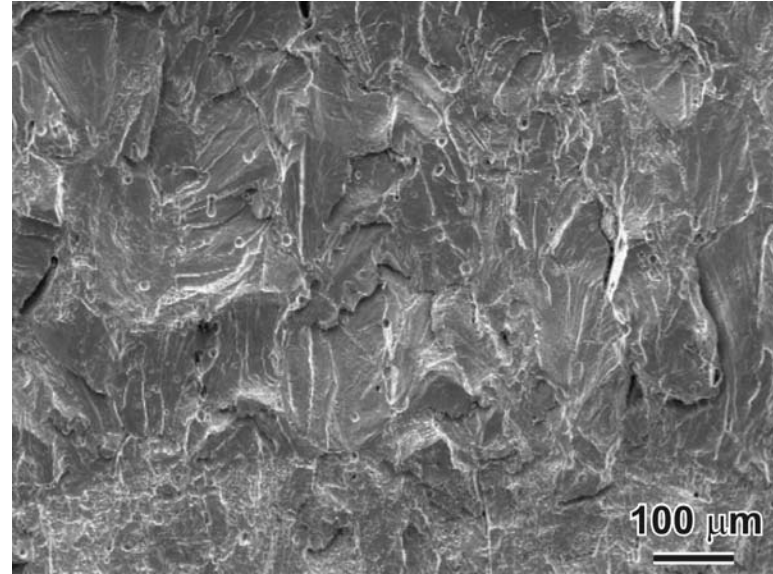




Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW

Region "B"
250°C APW with 1 ppm O₂
500s rise / 500 s fall

Continuous cycling with a 500s rise time
produced TG faceting,
in direct contrast with the IG cracking
observed in its sensitized counterpart.





Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW

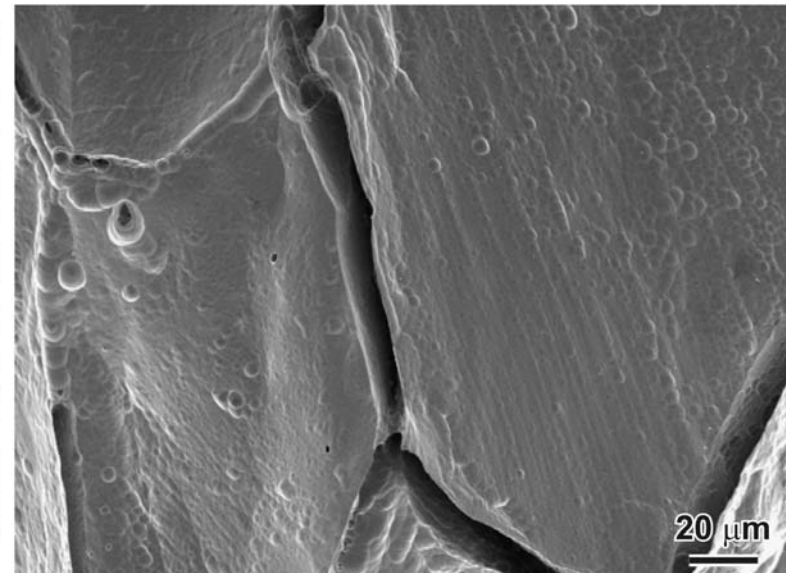
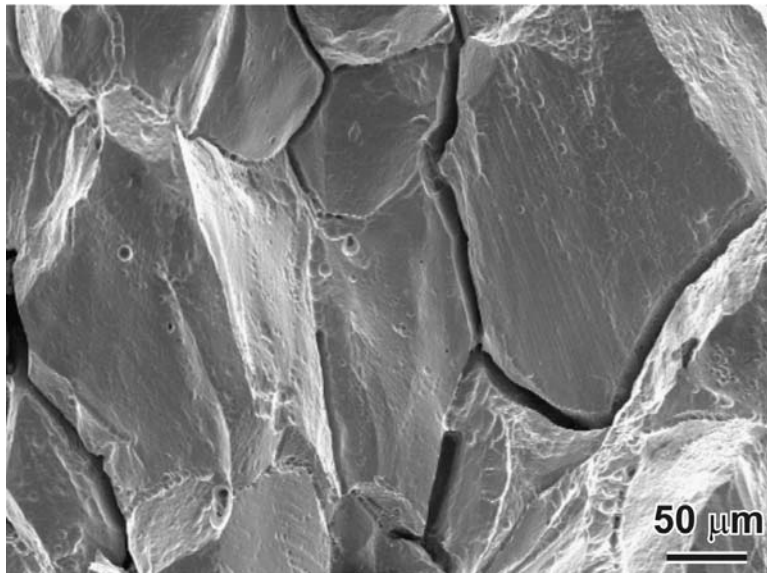
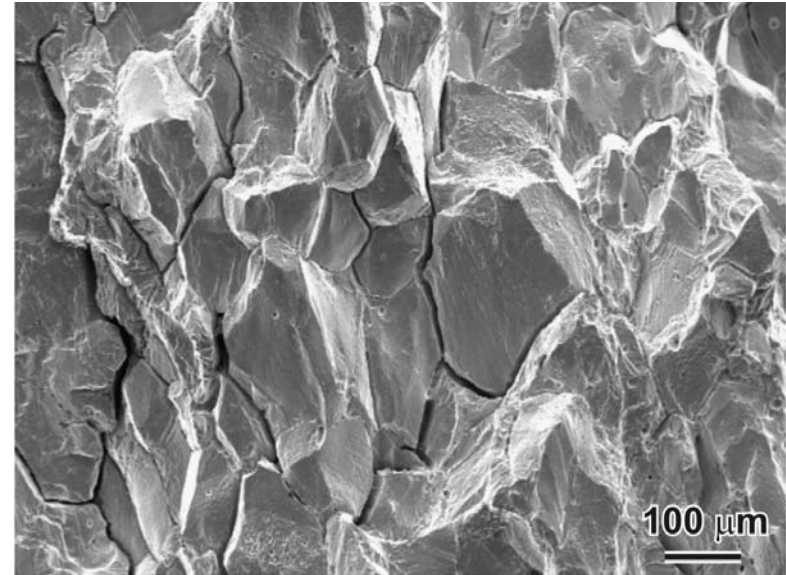
Region "C2"

250°C APW with 1 ppm O₂

500s rise / 9000s hold / 500 s fall

IGSCC is dominant.

Evidence of secondary IG cracking.





Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C DPW

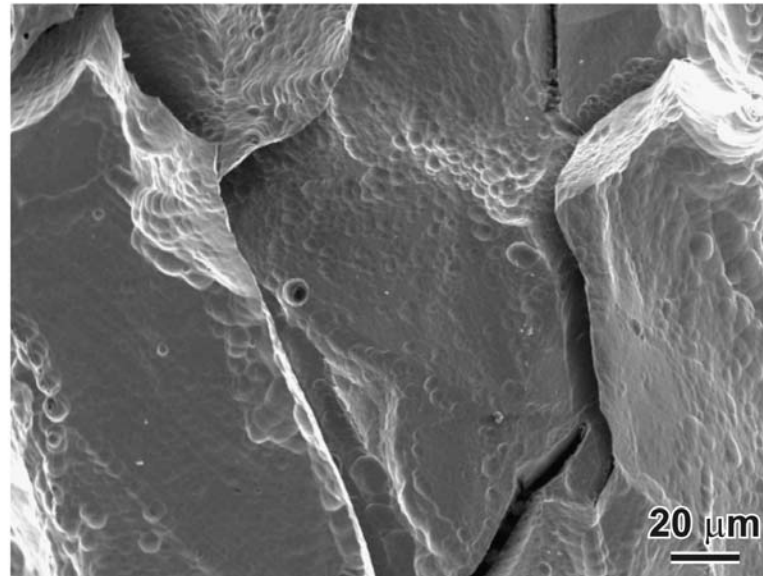
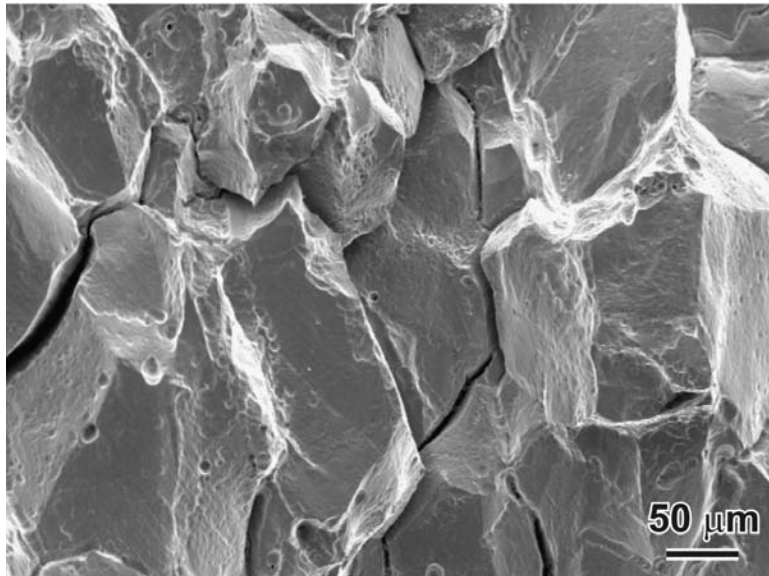
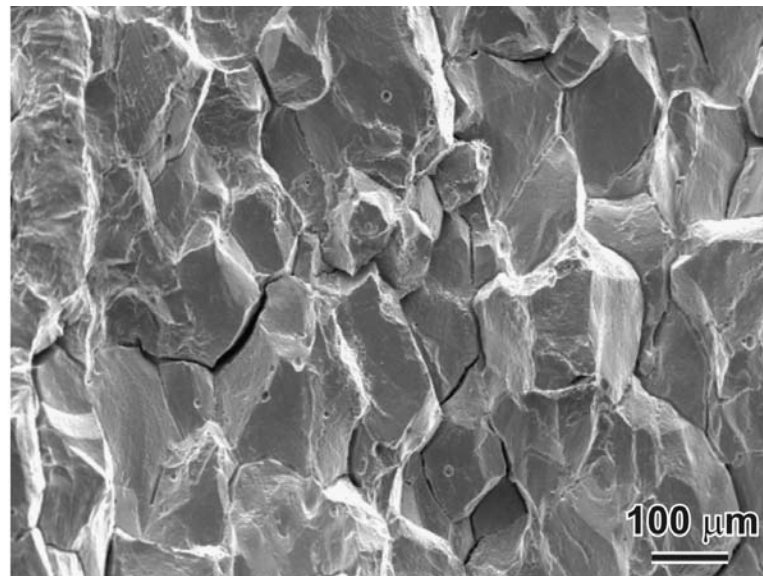
Region "C3"

250°C DPW with 25 cc H₂/kg H₂O
500s rise / 9000s hold / 500 s fall

IGSCC is dominant.

Evidence of secondary IG cracking.

Evidence of unbroken ligaments.





20% Cold Rolled 304 SS (CT-2CR)

Examination of corrosion products on the fracture surface revealed that the cracking mode in 250°C DPW was IG.

Gradient in ferrite crystal size was consistent with exposure times in APW & DPW:

<u>Region</u>	<u>Size of "Largest" Ferrite Crystals</u>
"B" (TG band)	20 – 40 μm
"C1" (just beyond TG band)	10 – 20 μm
"C2" (middle of IG)	~5 μm
"C3" (end of IG - DPW)	~2 μm
"C3" (IG finger beyond overall IG crack- DPW)	<1 μm

Gradient in "apparent" oxide thickness in between large ferrite crystals was consistent with exposure times in APW & DPW

(based on EDS X-ray analysis & difficulty in focusing on fracture surface).



20% Cold Rolled 304 SS (CT-2CR)

Region "B"

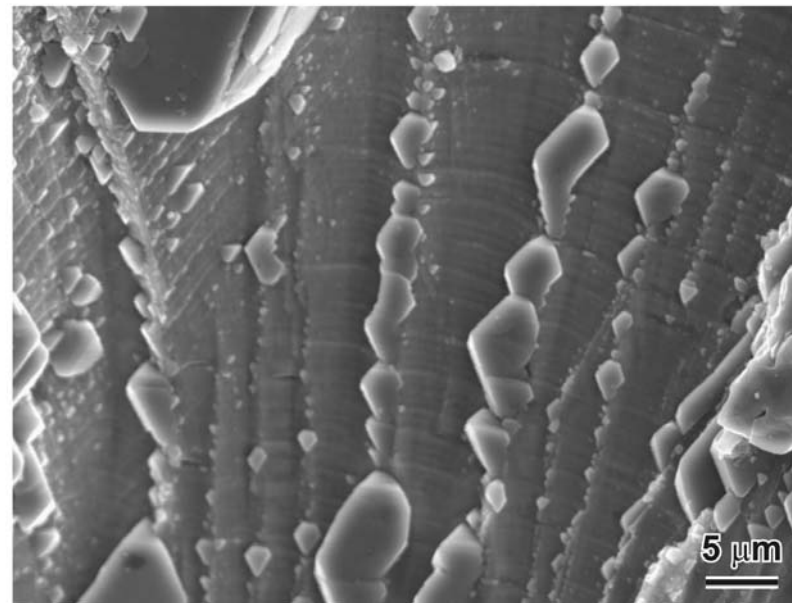
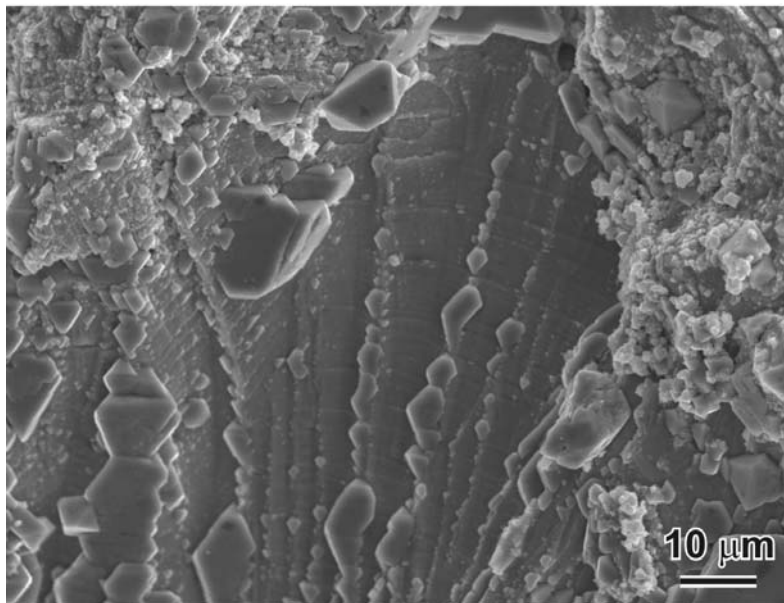
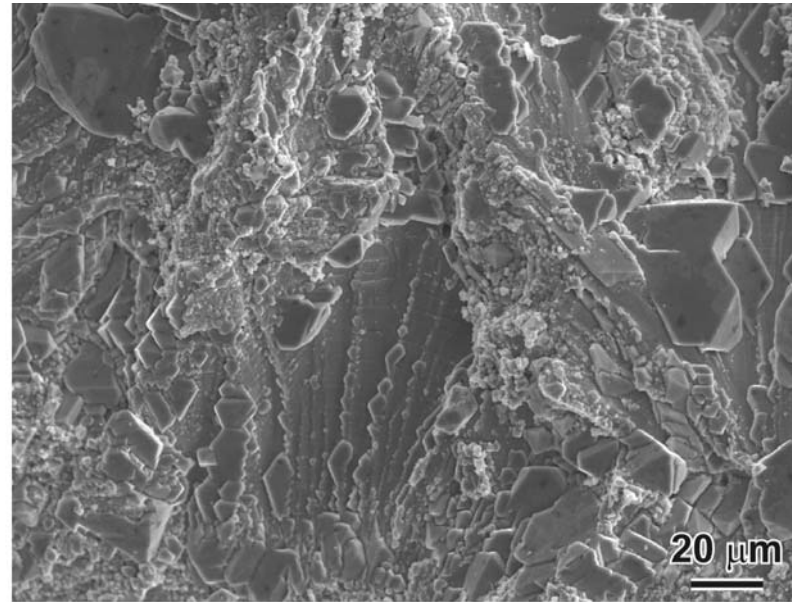
250°C APW: 1 ppm O₂

500s rise / 500s fall

TG facets w/ parallel fracture markings;
fatigue striations & slip offsets

Most regions are covered with a thick
oxide film and massive crystals.

Largest crystals are 20 – 40 μm.





20% Cold Rolled 304 SS (CT-2CR)

Region "C1"

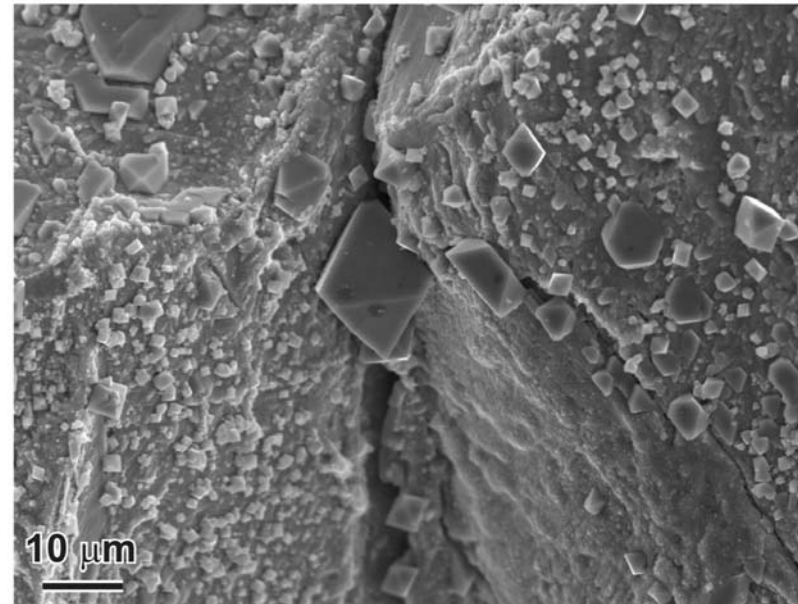
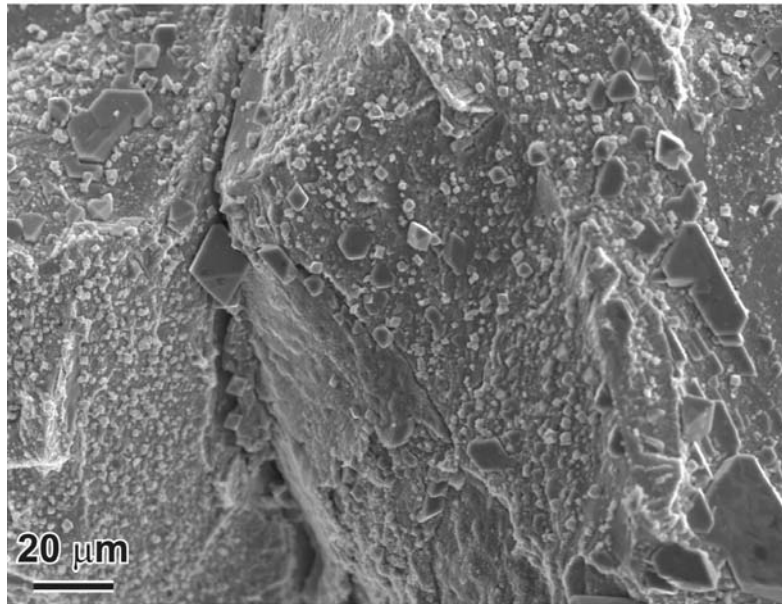
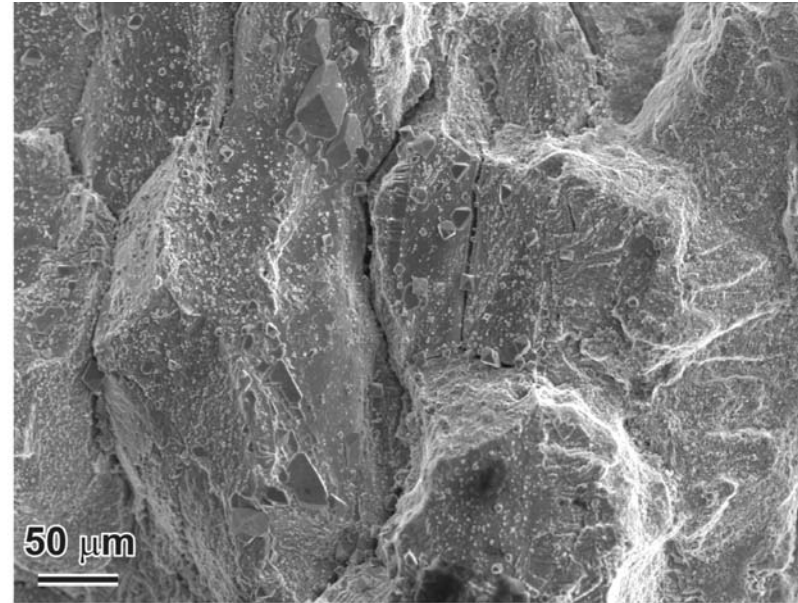
250°C APW: 0.2 ppm O₂

500s rise/9000s hold/500s fall

IGSCC is dominant.

IG faces are covered with a thick oxide film and large crystals.

Largest crystals are 10 - 20 μm.





20% Cold Rolled 304 SS (CT-2CR)

Region "C1"

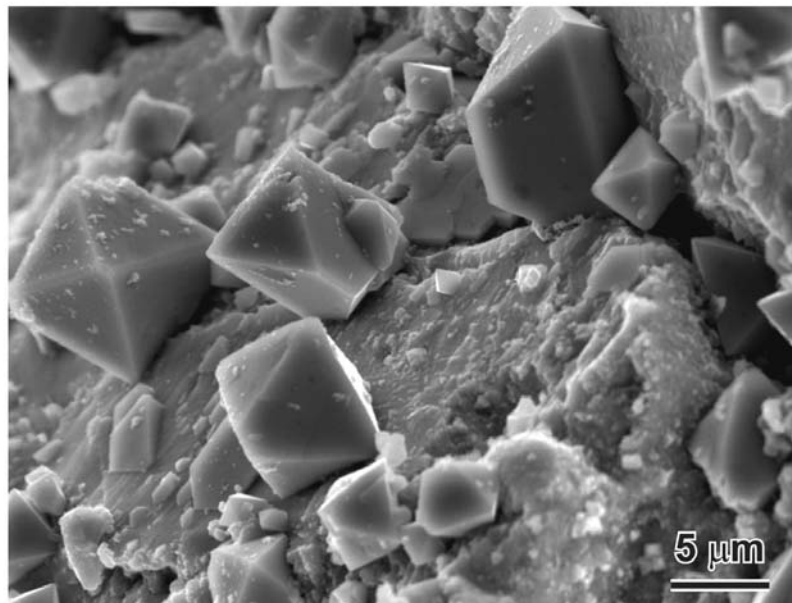
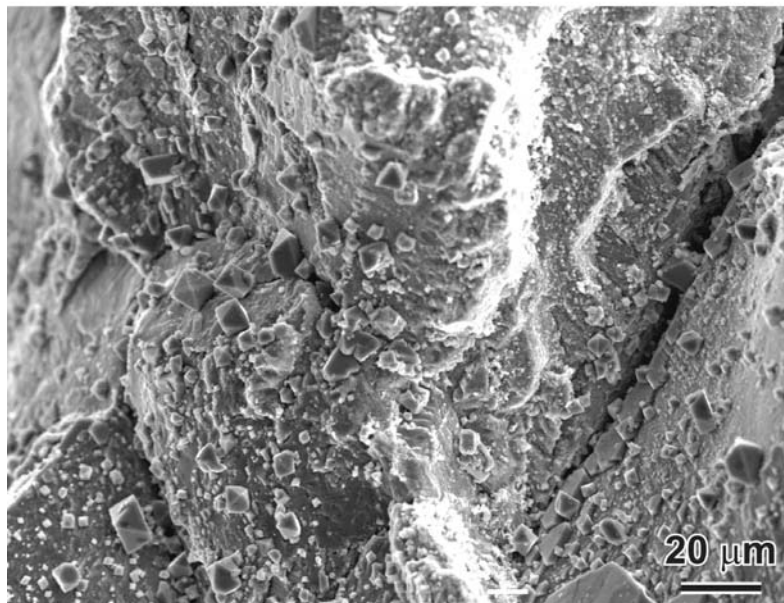
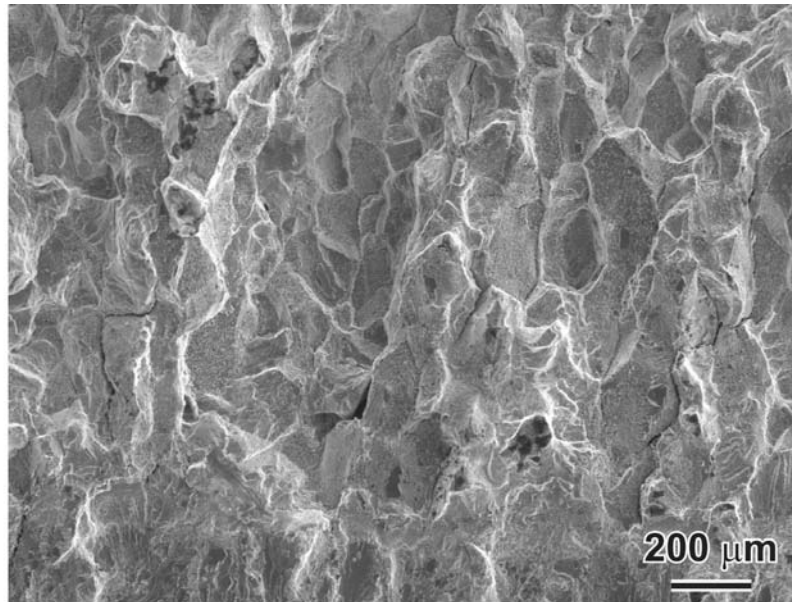
250°C APW: 0.2 ppm O₂

500s rise/9000s hold/500s fall

IGSCC is dominant, although there is evidence of TG islands.

IG faces are covered with a thick oxide film and large crystals.

Largest crystals are 10 - 20 μm .





20% Cold Rolled 304 SS (CT-2CR)

Region "C2"

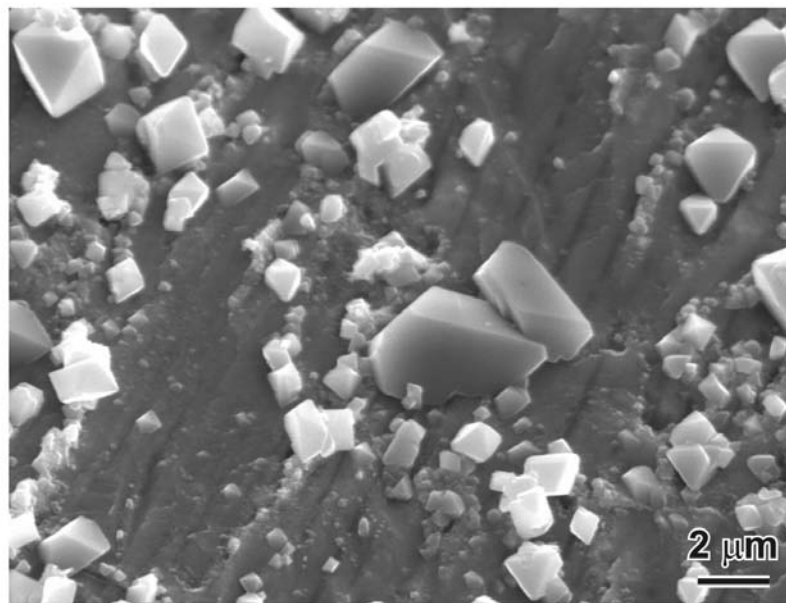
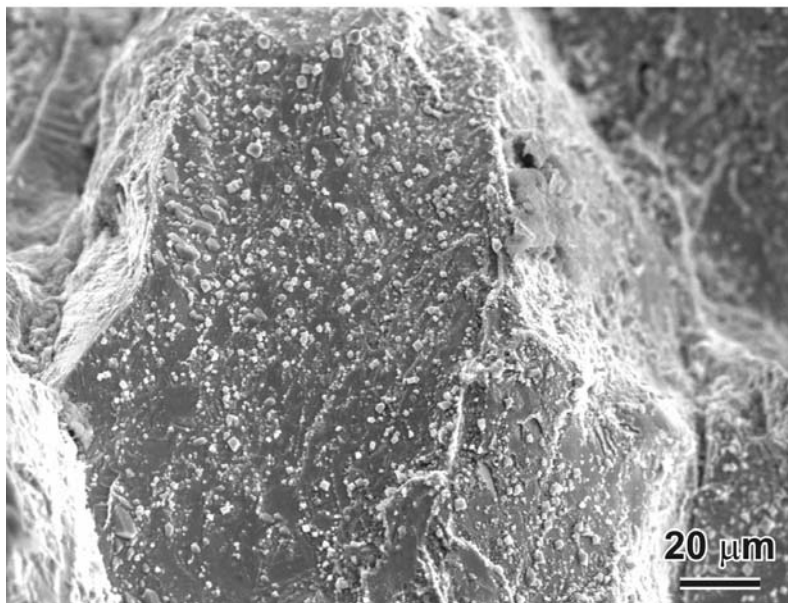
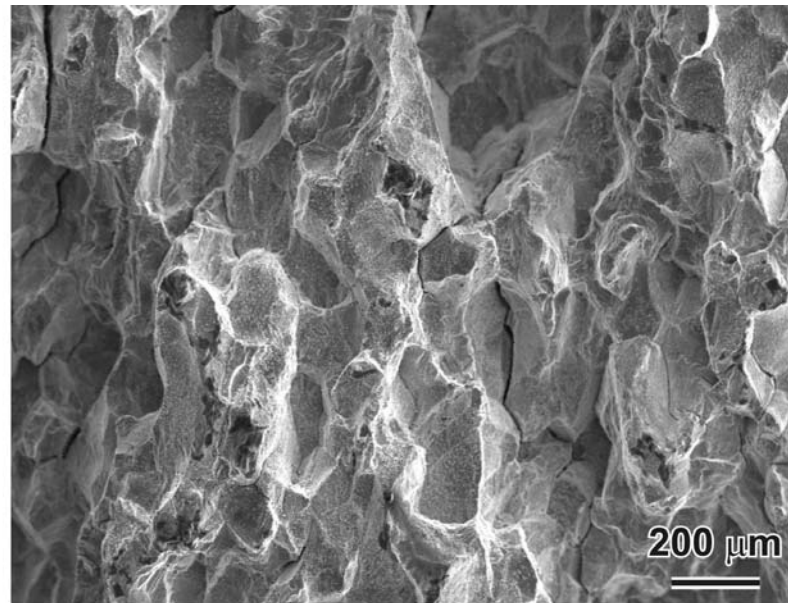
250°C APW: 1 ppm O₂

500s rise/9000s hold/500s fall

IGSCC is dominant, although there is evidence of TG islands.

IG faces are covered with moderate thick oxide film and large crystals.

Largest crystals are ~5 μm .





20% Cold Rolled 304 SS (CT-2CR)

Region "C3"

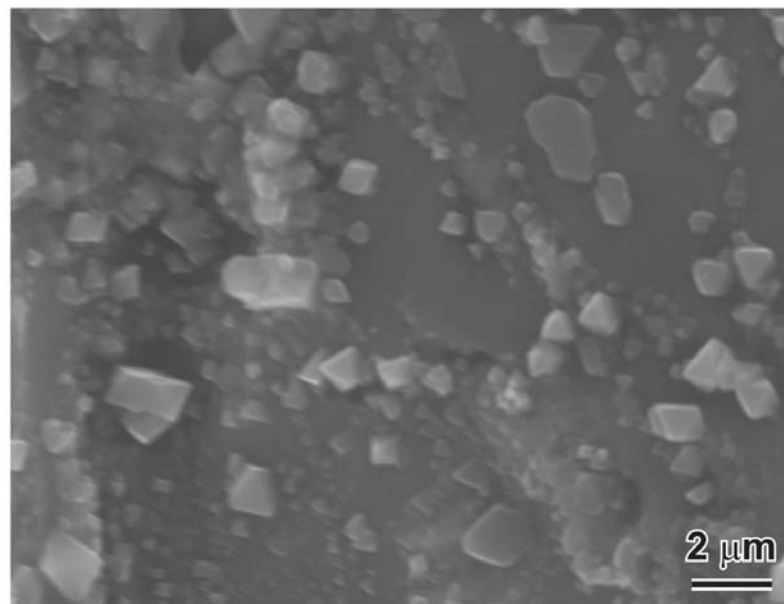
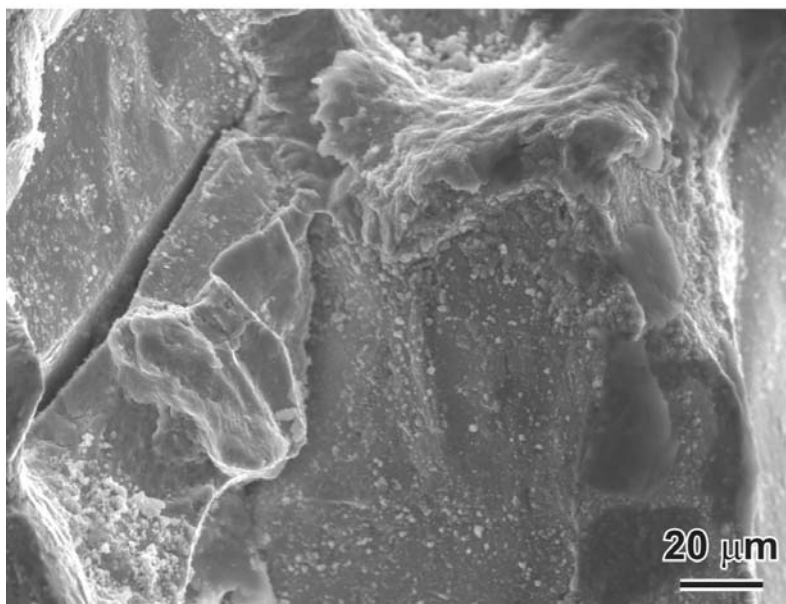
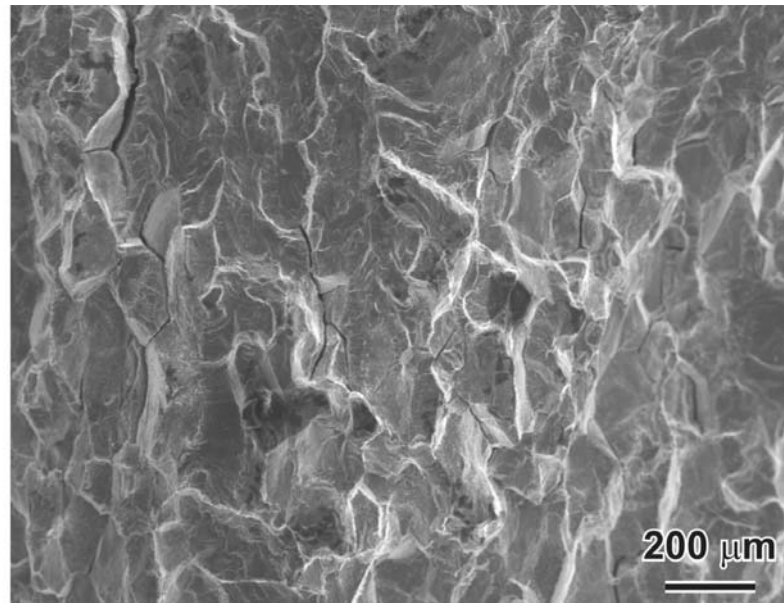
250°C DPW: 25 cc H₂/kg H₂O

500s rise / 9000s hold / 500s fall

IGSCC is dominant, although there is evidence of TG islands.

IG faces are covered with a thinner oxide film and micron-size crystals.

Largest crystals are ~2 μm.





20% Cold Rolled 304 SS (CT-2CR)

Region "C3" IG CRACK TIP

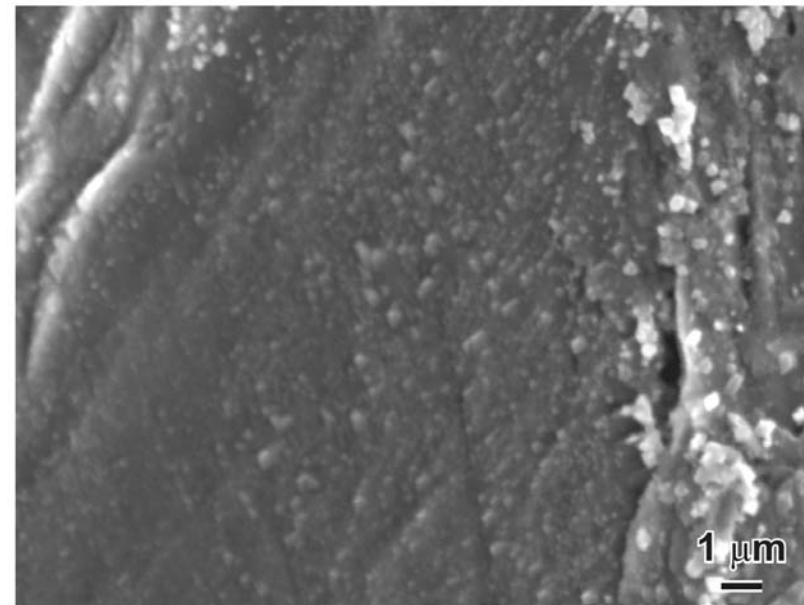
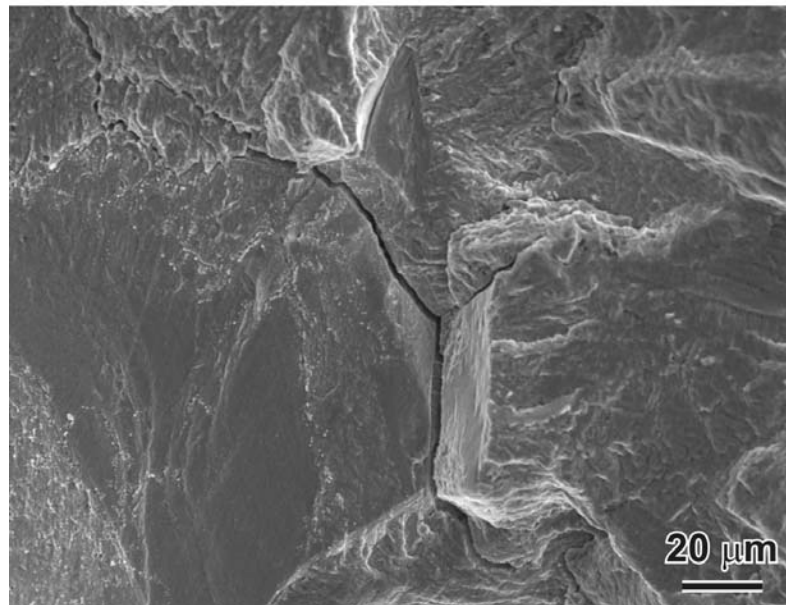
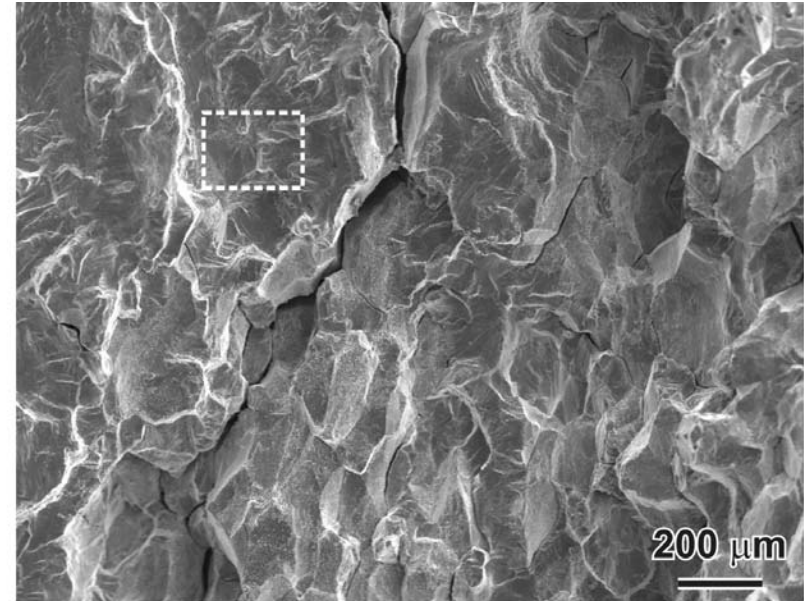
250°C DPW: 25 cc H₂/kg H₂O

500s rise/9000s hold/500s fall

IG finger well beyond IGSCC.

IG faces are covered with a thin oxide film and submicron-size crystals.

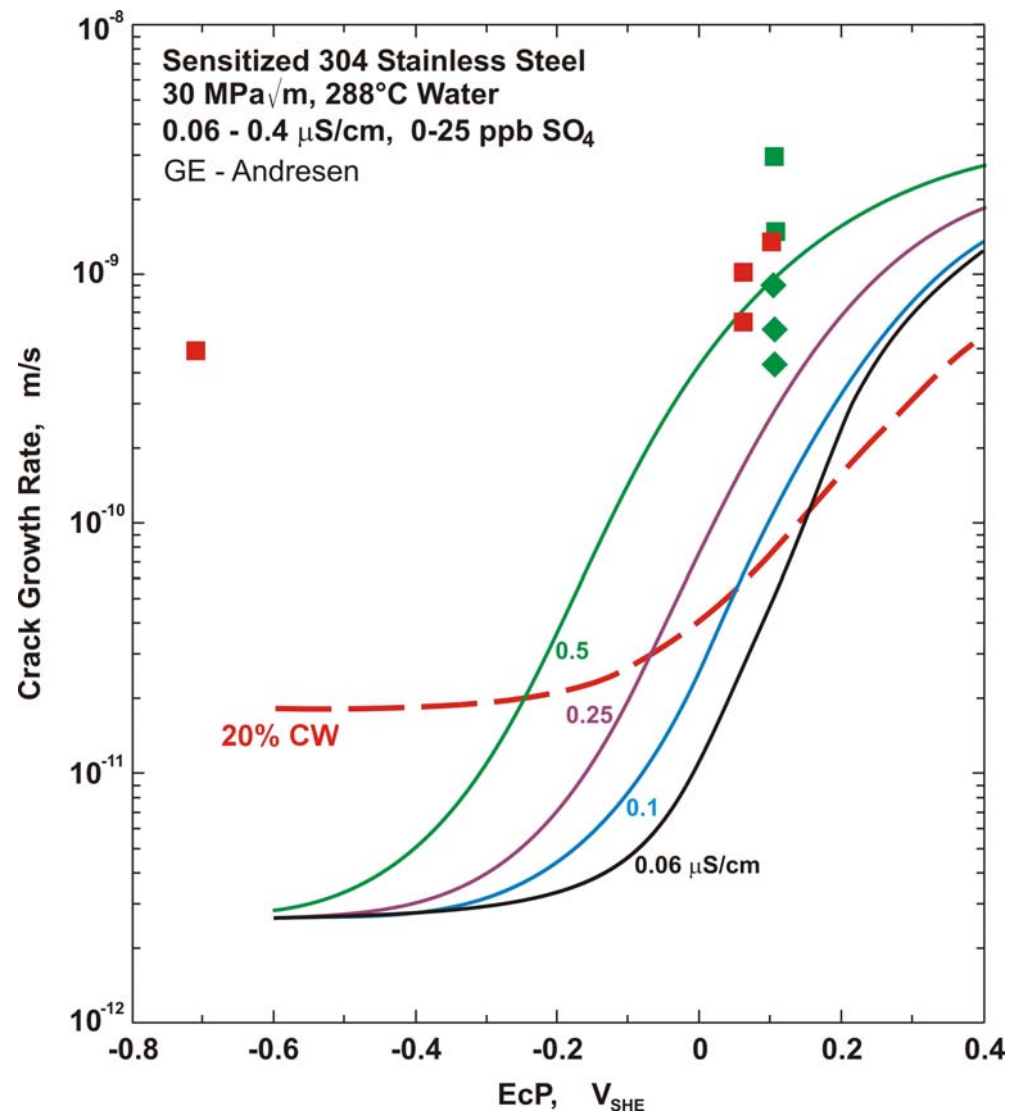
Largest crystals are <1 μm.





Comparison of current CGRs at 250°C with previously generated CGRs (GE - Andresen).

- Fully Sensitized 304SS
200 ppb SO₄
34-40 MPa√m
- ◆ Heavily Sensitized 304SS
200 ppb SO₄
29-35 MPa√m
- 20% Cold Rolled 304SS
~55 MPa√m





KEY FINDINGS FROM SCC SCOPING TESTS

Sensitized 304 SS in 75°C APW with 7 ppm O₂ and 200 ppb SO₄:

- Continuous cycling (500s / 5000s rise) produced both TG faceting & IGSCC.
- No measurable crack extension occurred with a 9000s hold.

Sensitized 304 SS in 250°C APW with 1 ppm O₂ and 200 ppb SO₄:

- Continuous cycling with 500s rise produced IG cracking.
- Rapid IGSCC (1-10 mils/day) occurred when a 9000s hold was introduced.

Sensitized 304 SS in 250°C DPW with 30 cc H₂/kg H₂O & 200 ppb SO₄:

- Transition to deaerated & hydrogenated water caused an order of magnitude reduction in CGRs (~0.1 mil/day).

Cracking mode in DPW was predominantly TG.

20%CW 304 SS in 250°C APW / DPW

- Continuous cycling with 500s rise produced TG cracking.
- With a 9000s hold, IGSCC readily initiated in APW & continued in DPW.
IG crack was discontinuous & highly branched.



DIRECTION FOR FUTURE SCC TESTING OF 304 SS

*Conduct SCC Tests on **Sensitized & Cold Worked** 304 SS*

- Sensitized 304 SS exhibited low CGRs in 250°C DPW & the cracking mode was TG.
- 20% CW^{ed} 304 SS exhibited high CGRs in 250°C DPW & the cracking mode was IG.
- Future testing will emphasize **sensitized & 10% CW 304 SS**.

Anion Contamination

- Future tests will use an anion contaminant level of 200 ppb SO₄.

Reduce the Number of Test Phases Performed on Each Specimen

- It is important to correlate fracture surface morphology with each test phase.



DIRECTION FOR FUTURE SCC TESTING OF 304 SS (continued)

Initiate IGSCC in APW with Trapezoidal Waveforms prior to DPW Testing

- IGSCC successfully generated in APW by cycling with 500s rise/500s fall followed by 500s rise/9000s hold/500s fall. (R=0.7)
Patterned after Morton (KAPL) & Andresen (GE).
- Similar loading / environmental sequence will be used in future SCC tests.

Potential Importance of Cyclic and Ripple Loading in Promoting SCC

- Cyclic loading may contribute to SCC susceptibility.
- Future SCC testing will evaluate the effects of cyclic stresses.
- Constant load tests are also needed to determine if SCC occurs in the absence of cyclic loads.



Backup Slides



Fully Sensitized 304 SS (WLP2-10)

Phase 5

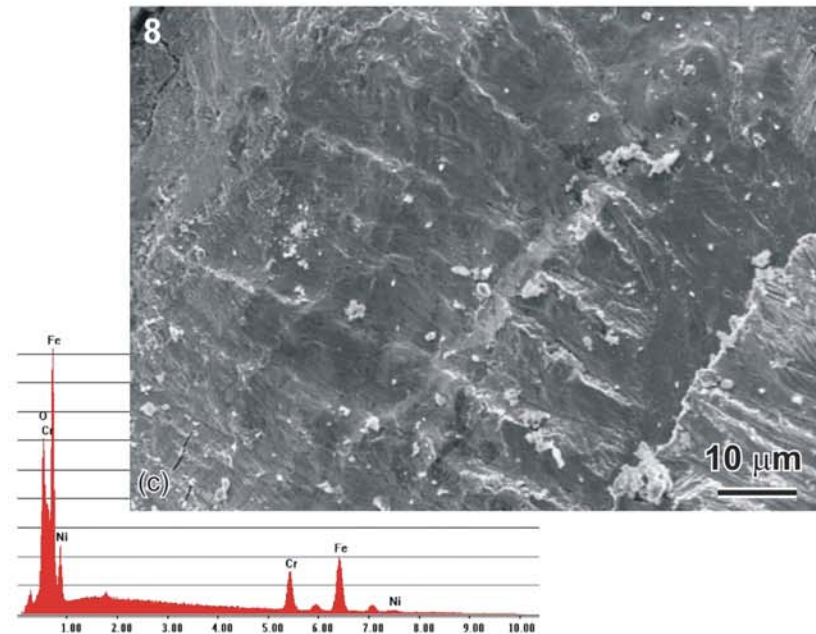
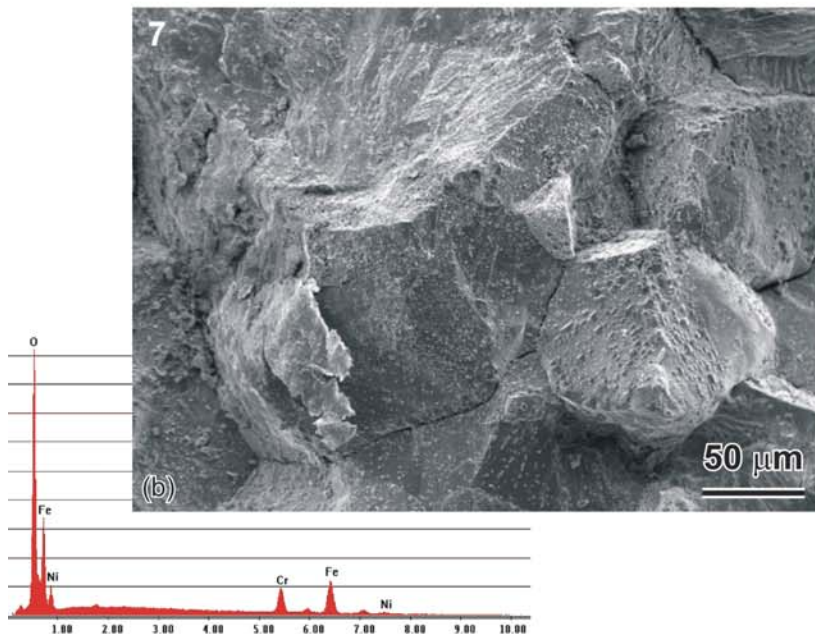
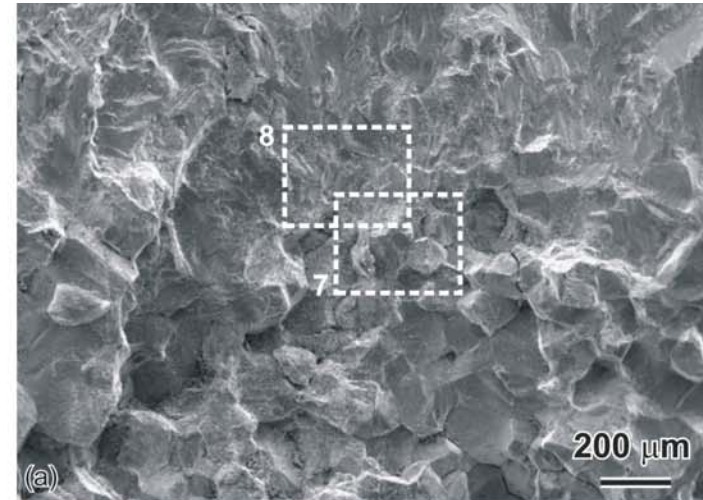
250°C APW / DPW with SO_4 .

500s rise / 9000s hold / 500s fall.

FEG-ESEM X-ray analysis was used to determine if cracking occurred in 250°C APW, 250°C DPW or 24°C air.

IG crack formed in APW had a thick oxide.

Broad TG facets formed in DPW had a thinner oxide.



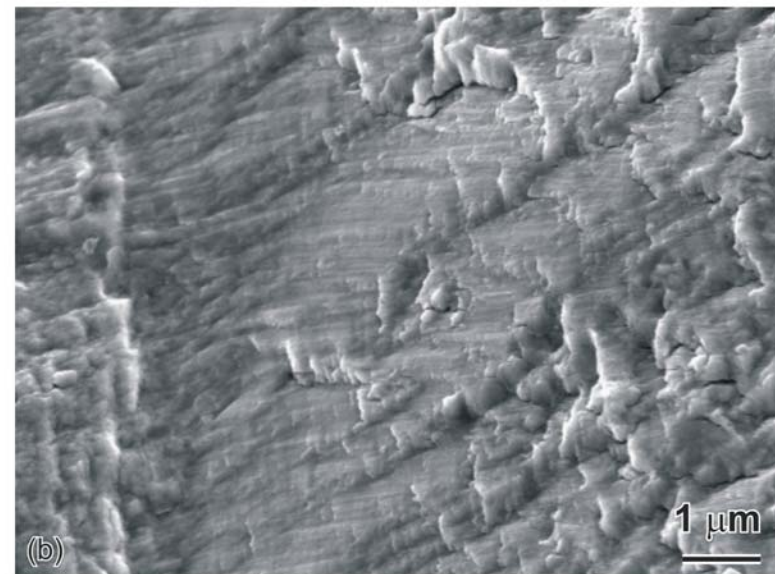
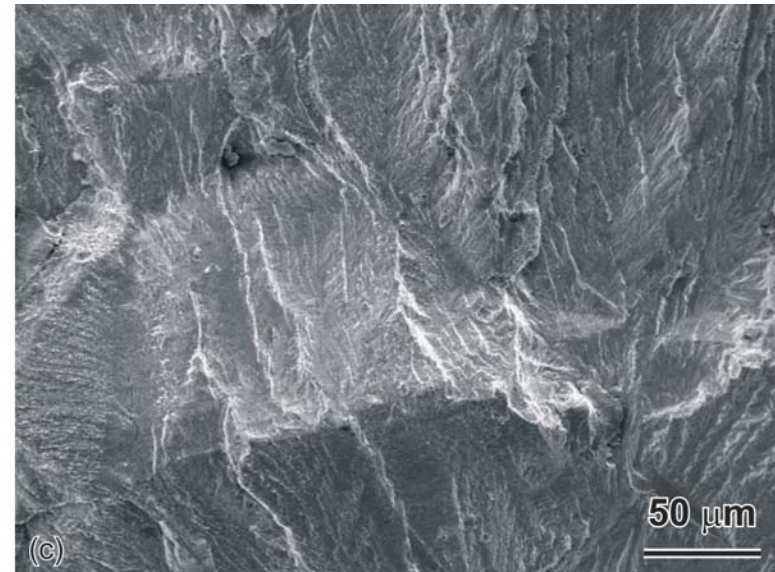
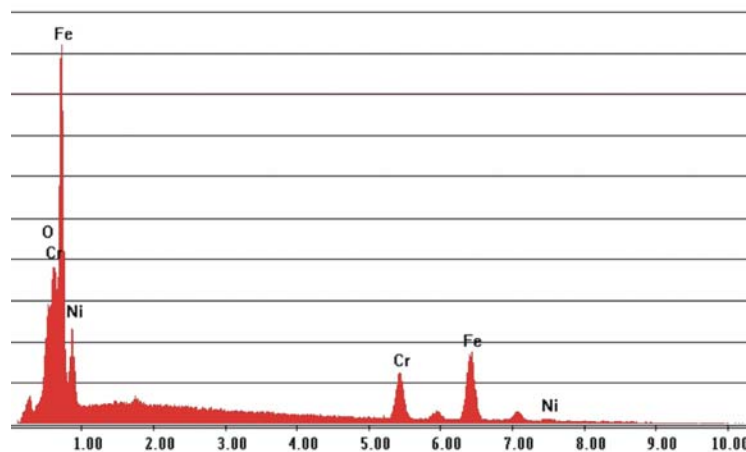


Fully Sensitized 304 SS (WLP2-10)

Post-test Fatigue Apart

FEG-ESEM X-ray analysis determined when cracking occurred (APW / DPW / Air).

Crystallographic facets formed during post-test fatigue apart contained a very thin oxide.





**Specimen CT-2CR
20% Cold Rolled 304 SS
Post-test Fatigue in Air**

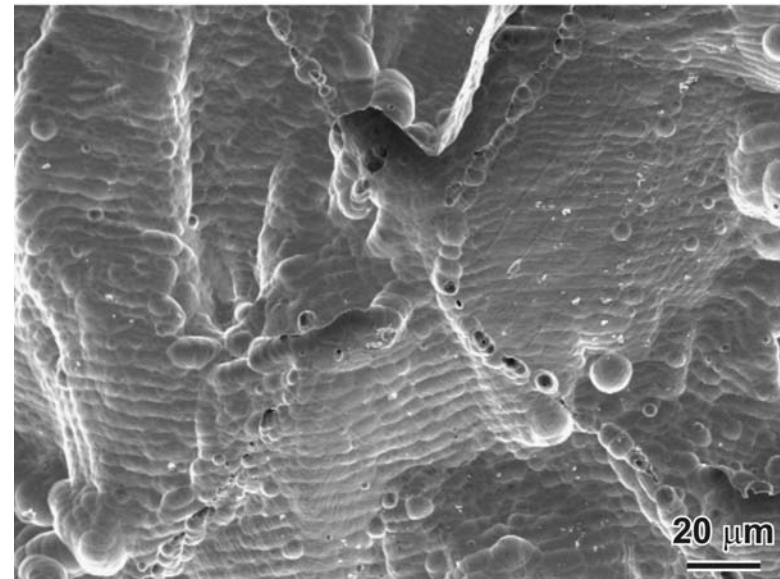
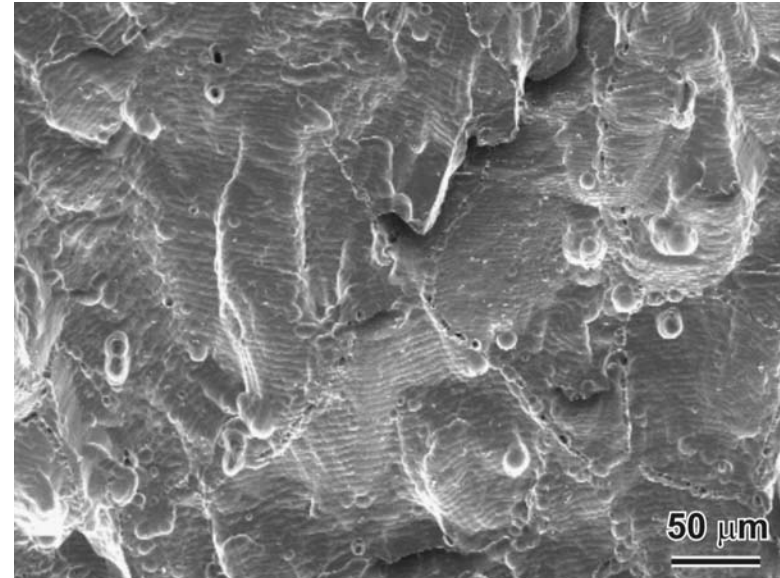
Region "Post"

24°C Air

Continuous Cycling

Classic fatigue striations.

ENDOX cleaning pitted the surface
of the 20% CW 304 SS,
in contrast with the lack of pitting
on the non-cold worked 304 SS.





20% Cold Rolled 304 SS
Tested in 250°C APW & 250/338°C DPW

In 338°C DPW, sustained IGSCC did not incubate from a TG fatigue precrack.

In 250°C APW (0.2 & 1 ppm O₂), IGSCC readily incubated from a TG precrack.

Rapid IGSCC continued when the water was deaerated and hydrogenated to 25 cc H₂/kg H₂O.



20% Cold Rolled 304 SS Specimen CT-1CR - Tested in 338°C DPW

Pre-crack was confined to two corner cracks where the notch intersected the side surfaces.



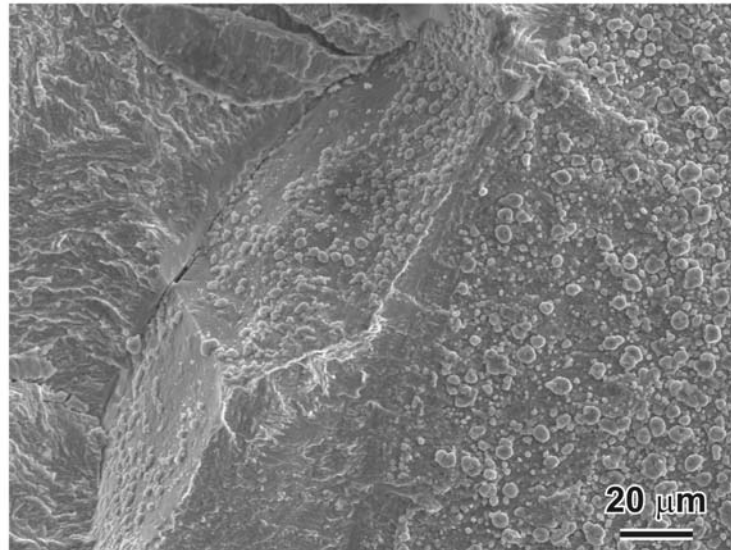
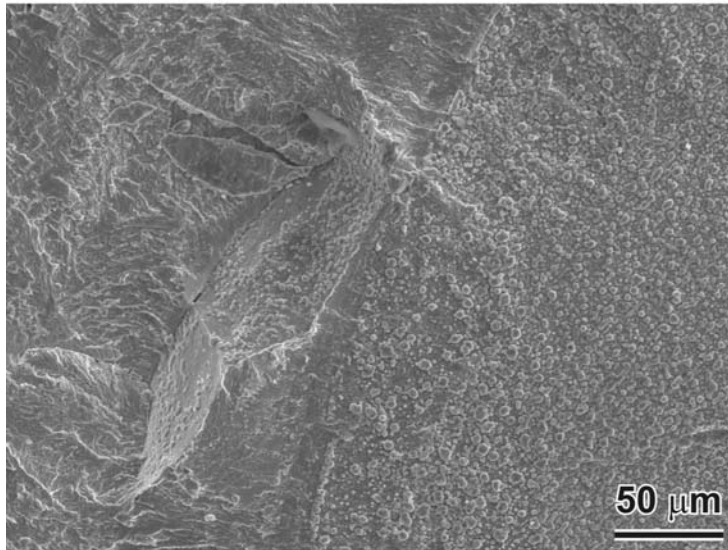
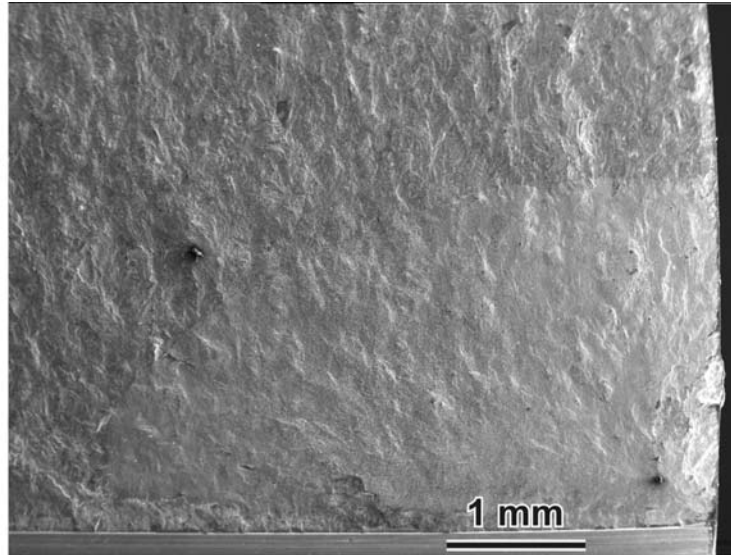


Specimen CT-1CR
20% Cold Rolled 304 SS
Tested in 338°C DPW

IGSCC was not sustained.

IGSCC was observed at a single isolated region along the precrack.

IGSCC was confined to two grain boundary faces.





20% Cold Rolled 304 SS Specimen CT-2CR Tested in 250°C APW / 250°C DPW

Initial SCC testing in 250°C APW produced IGSCC from two the corner precracks.

Specimen was re-precracked & tested in 250°C APW / 250°C DPW.

APW - 500s or 5000s rise

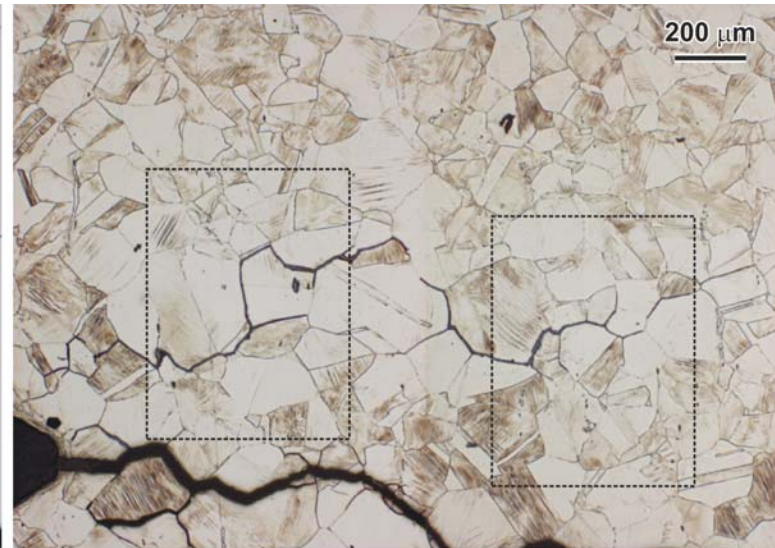
APW - 9000s hold

DPW - 9000s hold

Note unbroken ligaments in IG region, especially in IG region generated in DPW.

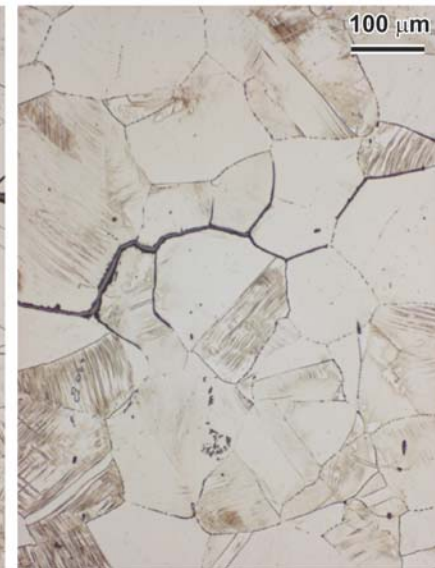
Metallographic Profile →





Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW

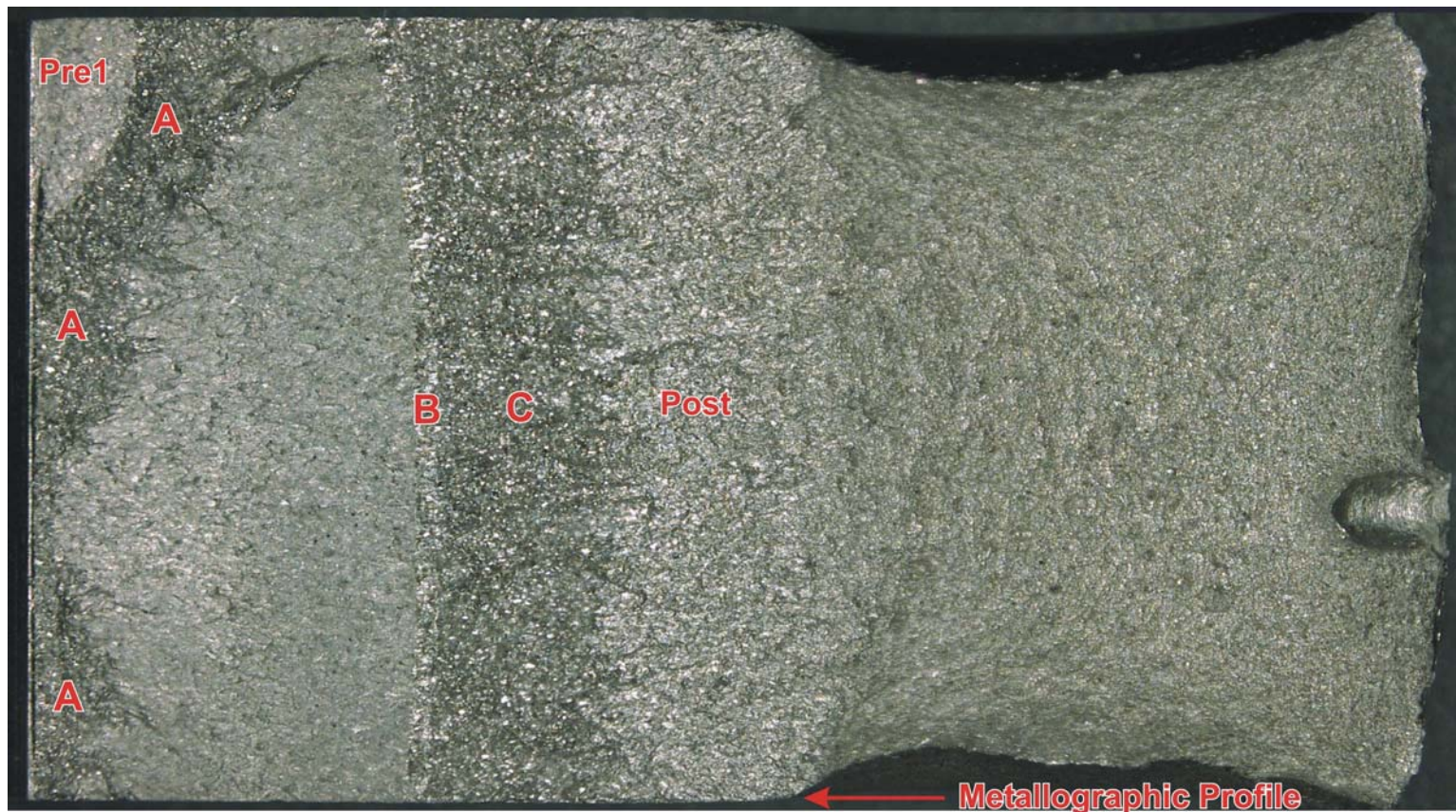
Branched IGSCC emanating from notch.





20% Cold Rolled 304 SS Specimen CT-2CR
Tested in 250°C APW / 250°C DPW

Initial SCC testing in 250°C APW produced IGSCC from two the corner precracks.
Specimen was re-precracked & tested in 250°C APW / 250°C DPW.



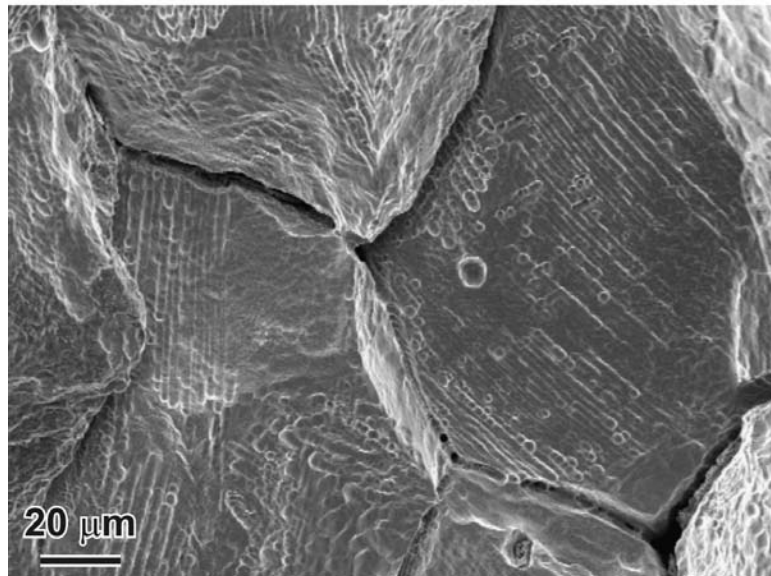
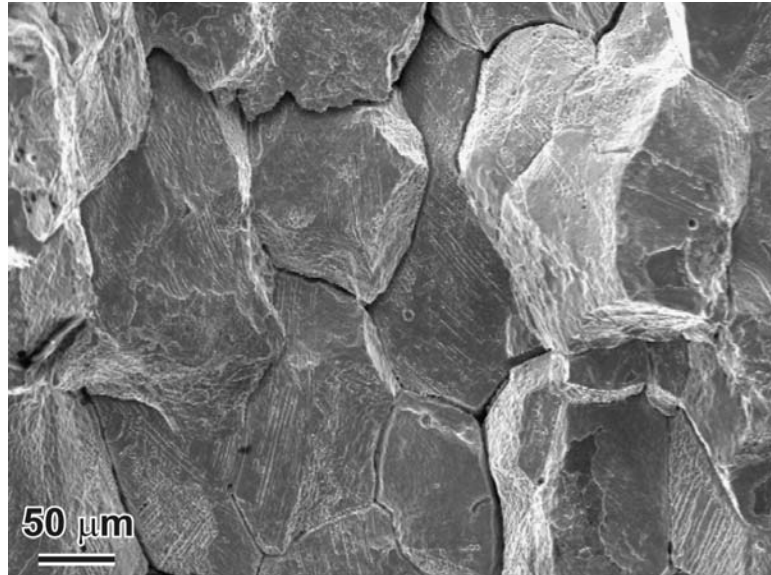


Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW

Region "A"

Classic IGSCC emanating from corner cracks.

Significant secondary IG cracking.





Fully Sensitized 304 SS (WLP2-10)

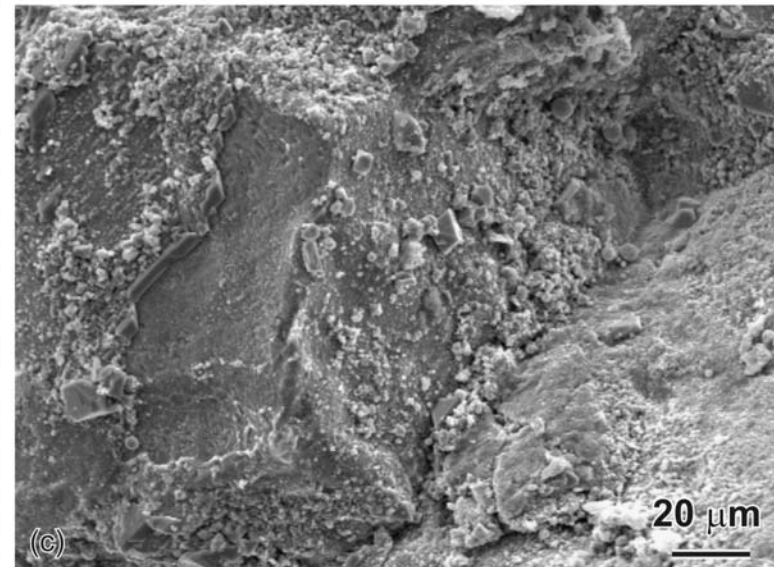
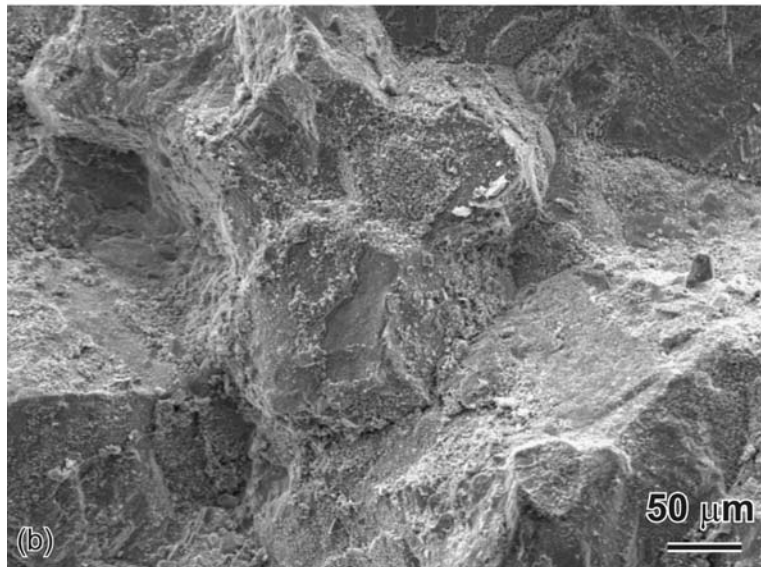
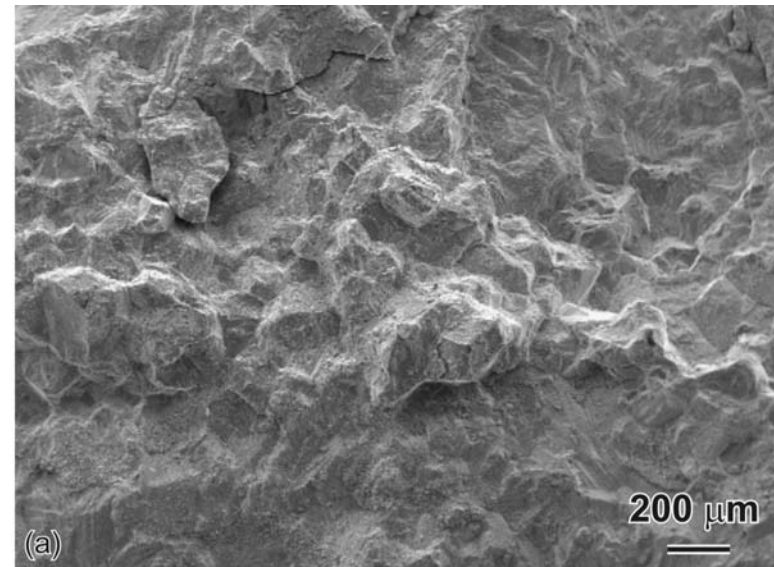
Phase 3

250°C APW with & without SO₄.

500s rise / 500s fall.

Narrow band of IGSCC.

Thick oxide & corrosion debris obscures fracture surface details.

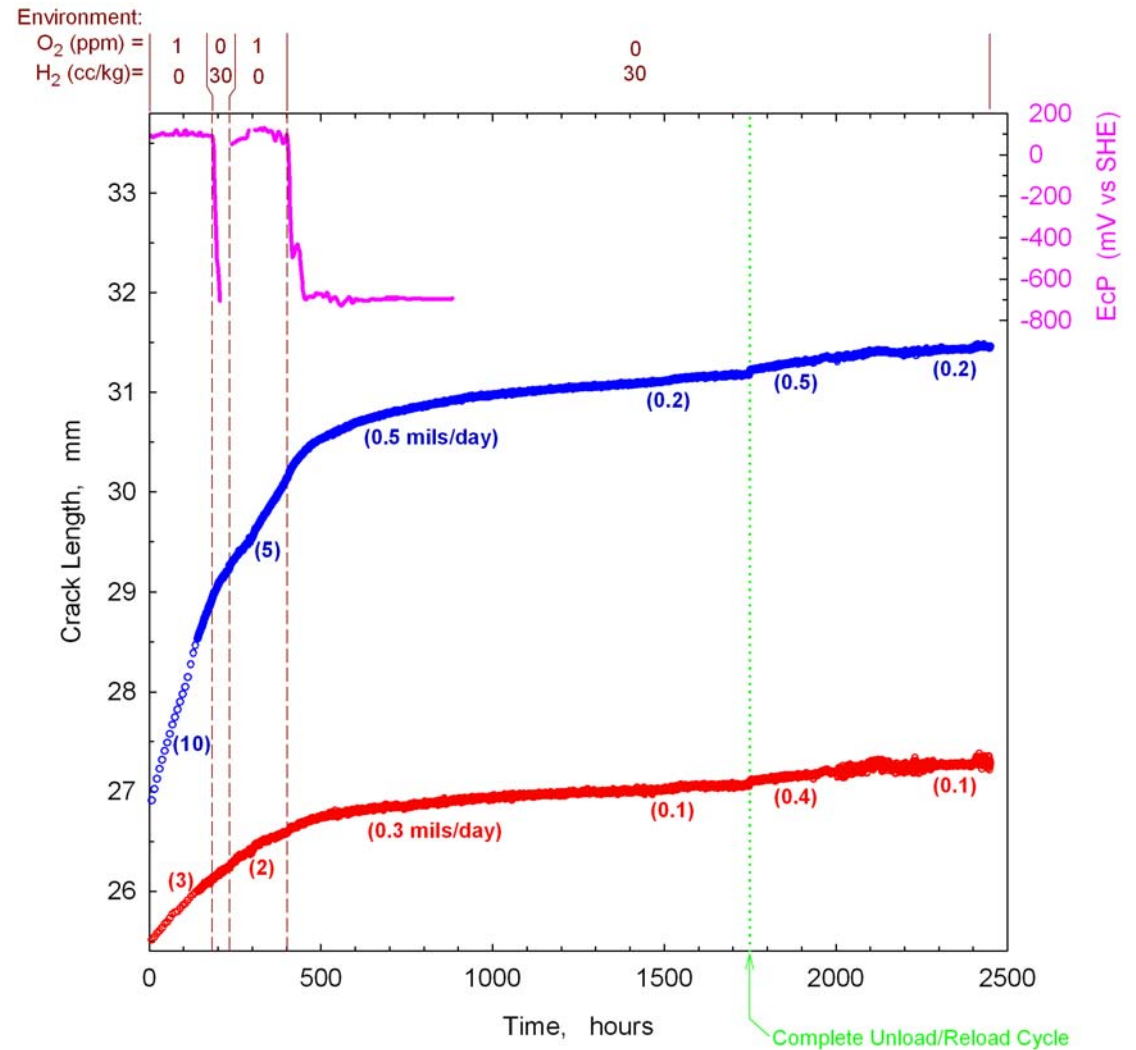




Phase 5 Testing
250°C APW with 200 ppb SO₄ /
250°C DPW with 200 ppb SO₄

500s rise / 9000s hold / 500s fall

- WLP2-6 (Heavily Sensitized)
- WLP2-10 (Fully Sensitized)





Phase 6a: 250°C APW w 200 ppb SO₄

500s rise / 500s fall

Phase 6b: 250°C APW w 200 ppb SO₄

500s rise / 9000s hold / 500s fall

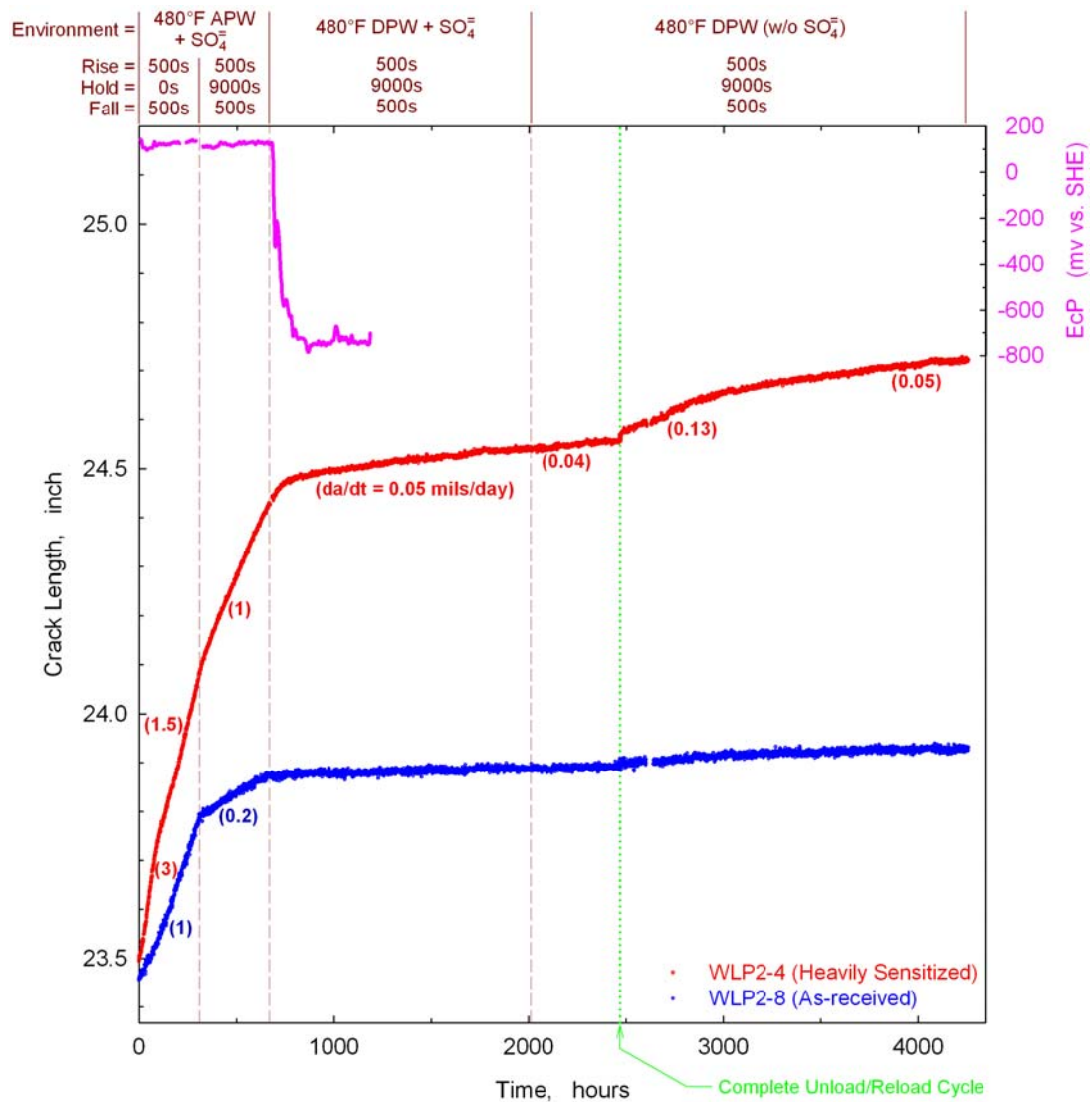
Phase 6c: 250°C DPW

w & w/o 200 ppb SO₄

500s rise / 9000s hold / 500s fall

Cracking mode for Heavily Sensitized Specimen WLP2-4 tested in 250°C APW was predominantly **IG**.

Cracking mode for As-Received Specimen WLP2-8 tested in 250°C APW was **TG**.





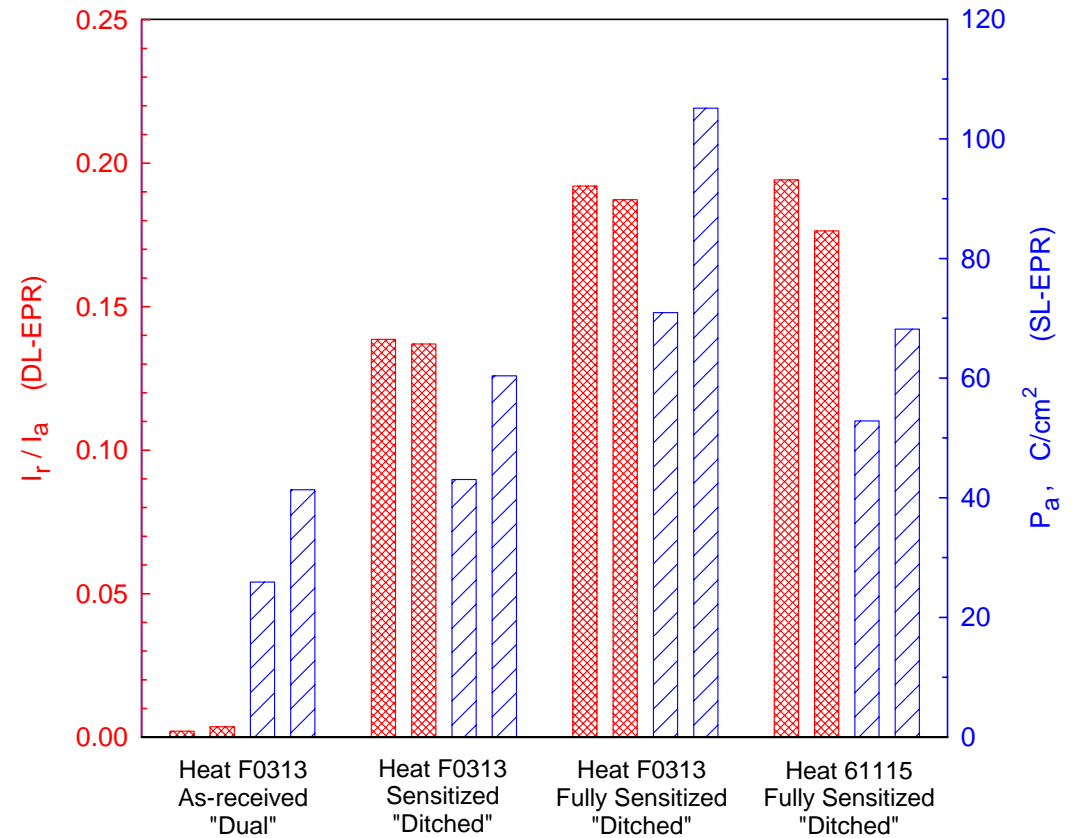
EPR Results for 304 SS Test Materials

DL-EPR

Activation/reactivation current ratio (I_r/I_a) is provided by red bars.

SL-EPR

Normalized charge (P_a) is provided by blue bars.





**Specimen CT-2CR
20% Cold Rolled 304 SS
Tested in 250°C APW**

Region "C1"

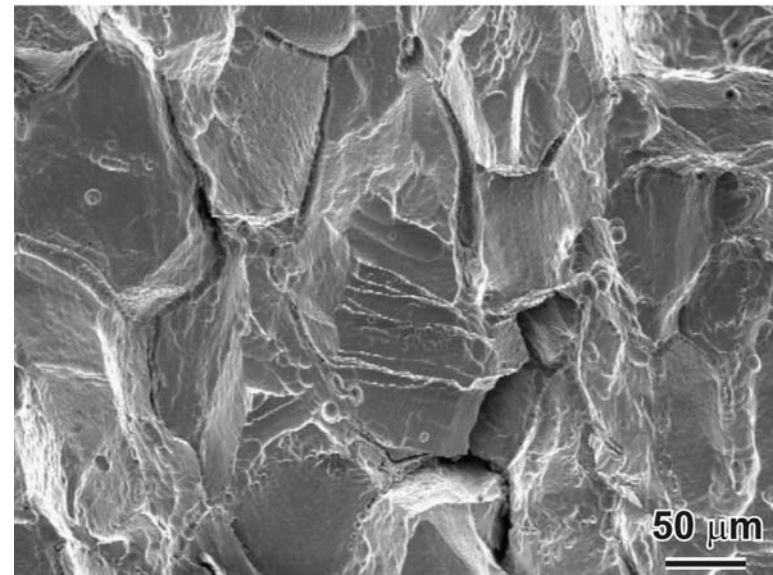
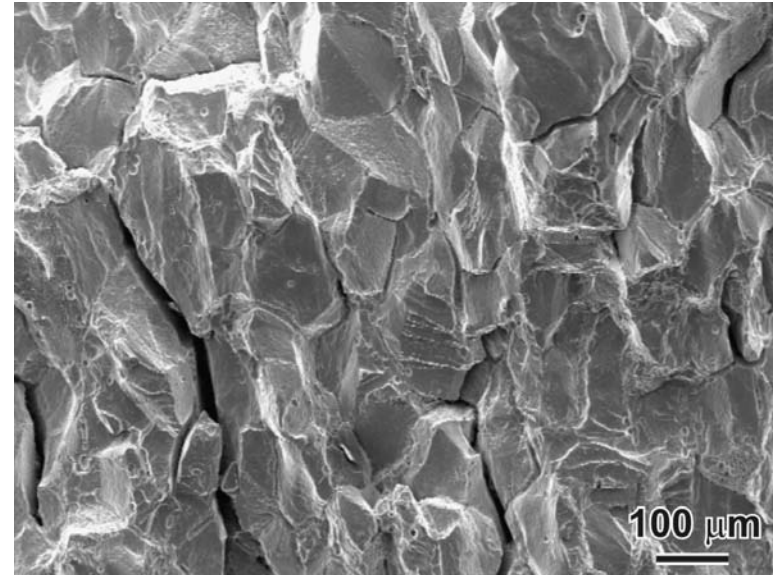
250°C APW with 0.2 ppm O₂

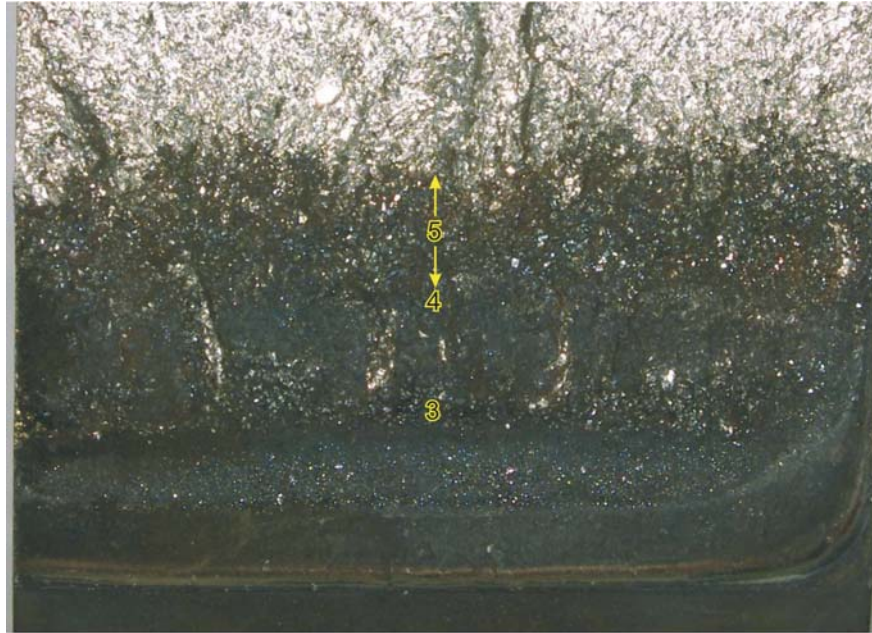
500s rise / 9000s hold / 500 s fall

IGSCC is dominant.

Evidence of secondary IG cracking.

Significant evidence of ligaments;
some ligaments eventually failed
during testing in water,
other ligaments failed during post-
test fatigue apart.





Heavily Sensitized 304 SS (WLP2-6)

Narrow IGSCC band during Phase 3 testing in 250°C APW with & without SO₄.

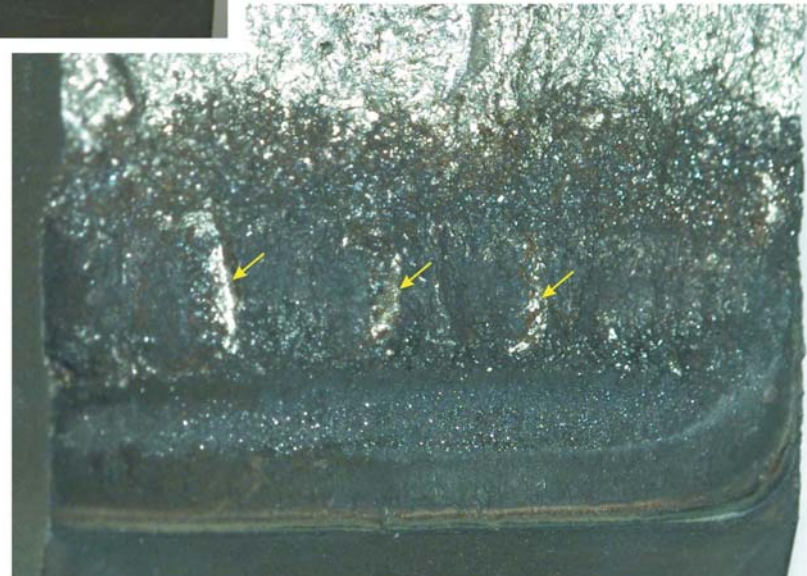
#3 - 500s rise / 500s fall.

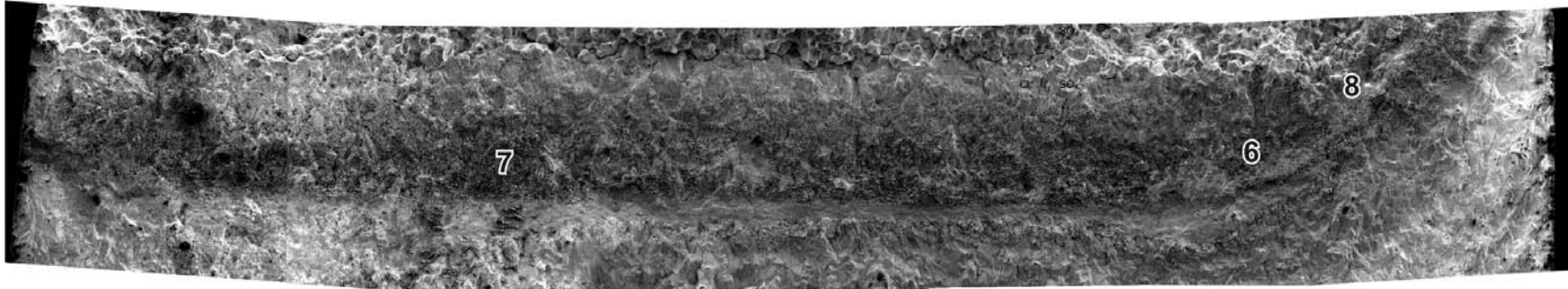
Extensive IGSCC during Phase 4/5 testing 250°C APW with SO₄.

#4 - 500s rise / 500s fall

#5 - 500s rise / 9000s hold / 500s fall.

Fracture surface was covered with a dark oxide and orange-red deposits.

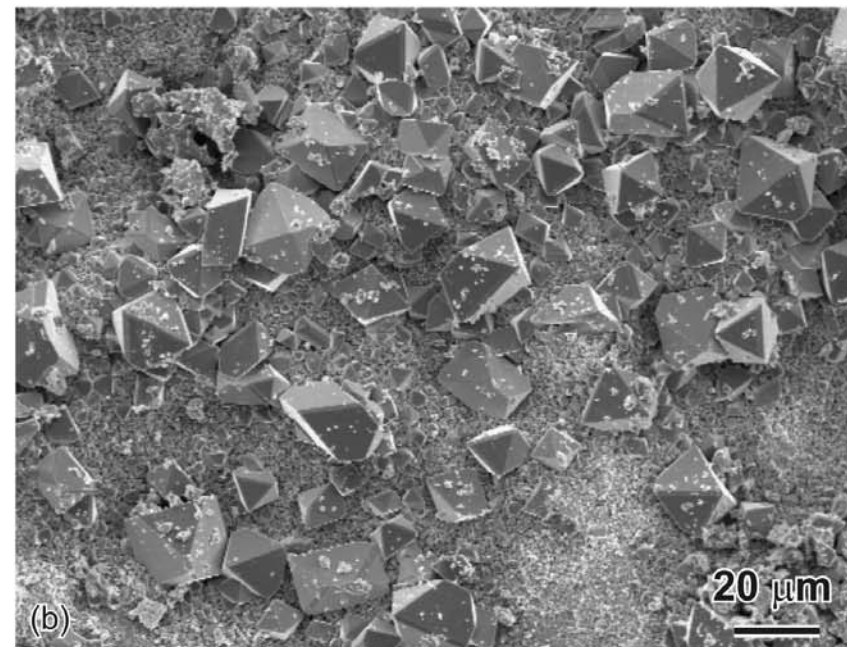




Heavily Sensitized 304 SS (WLP2-6)

Dark band associated with a crevice ring in the fatigue precrack region near the crack mouth.

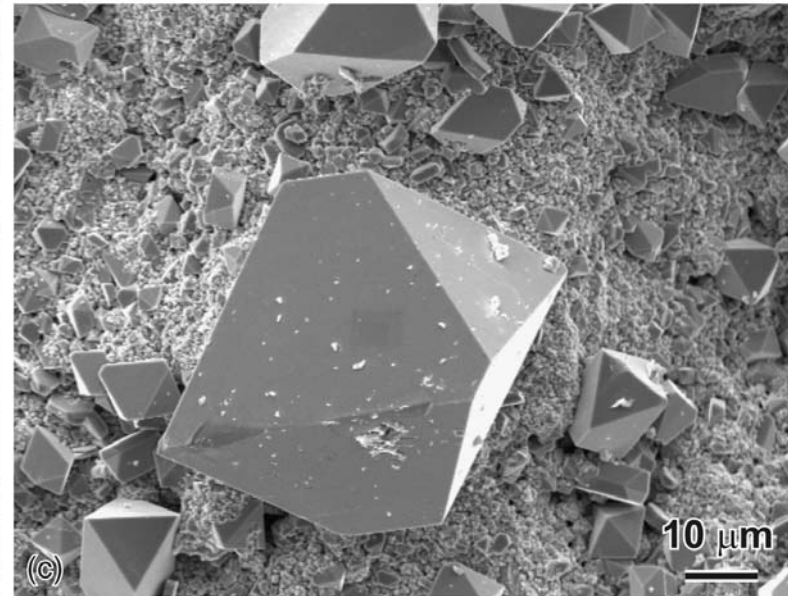
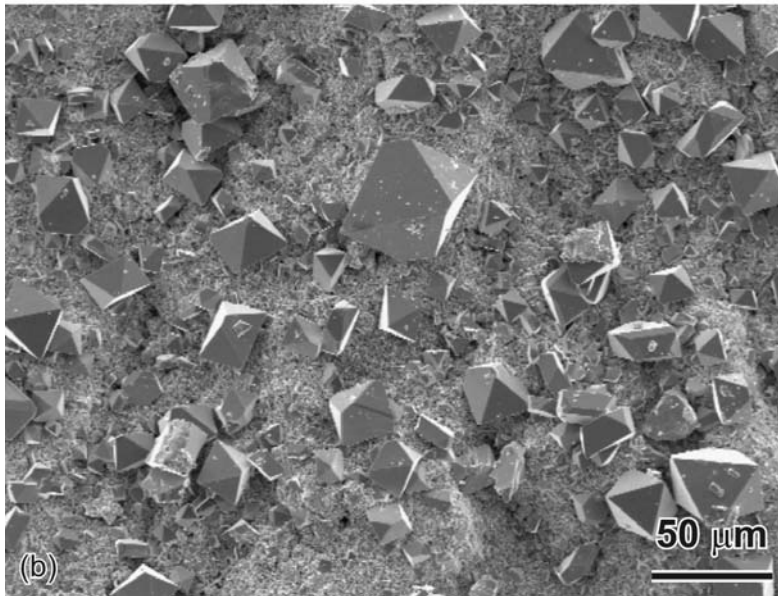
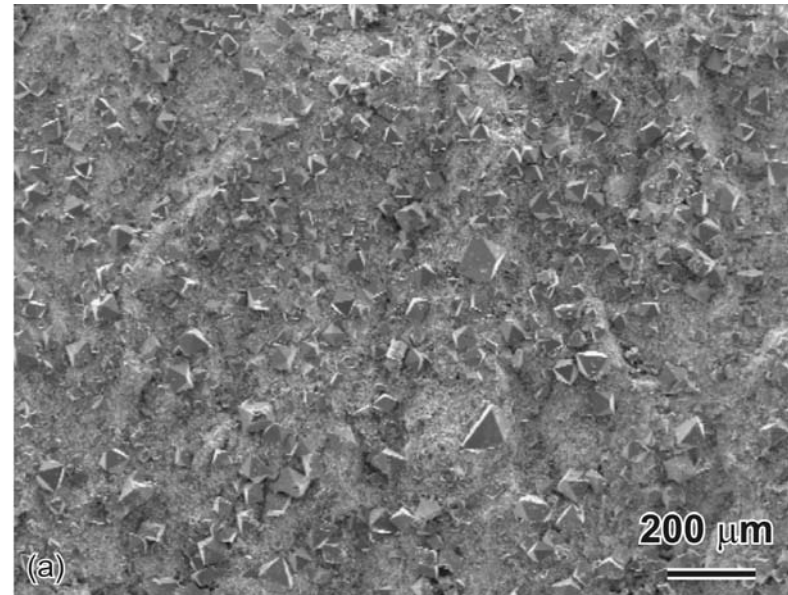
High density of large crystals in a dark band.





Heavily Sensitized 304 SS (WLP2-6)

High density of large crystals in a dark band near crack mouth.





20% Cold Rolled 304 SS (CT-2CR)
Tested in 250°C APW / DPW

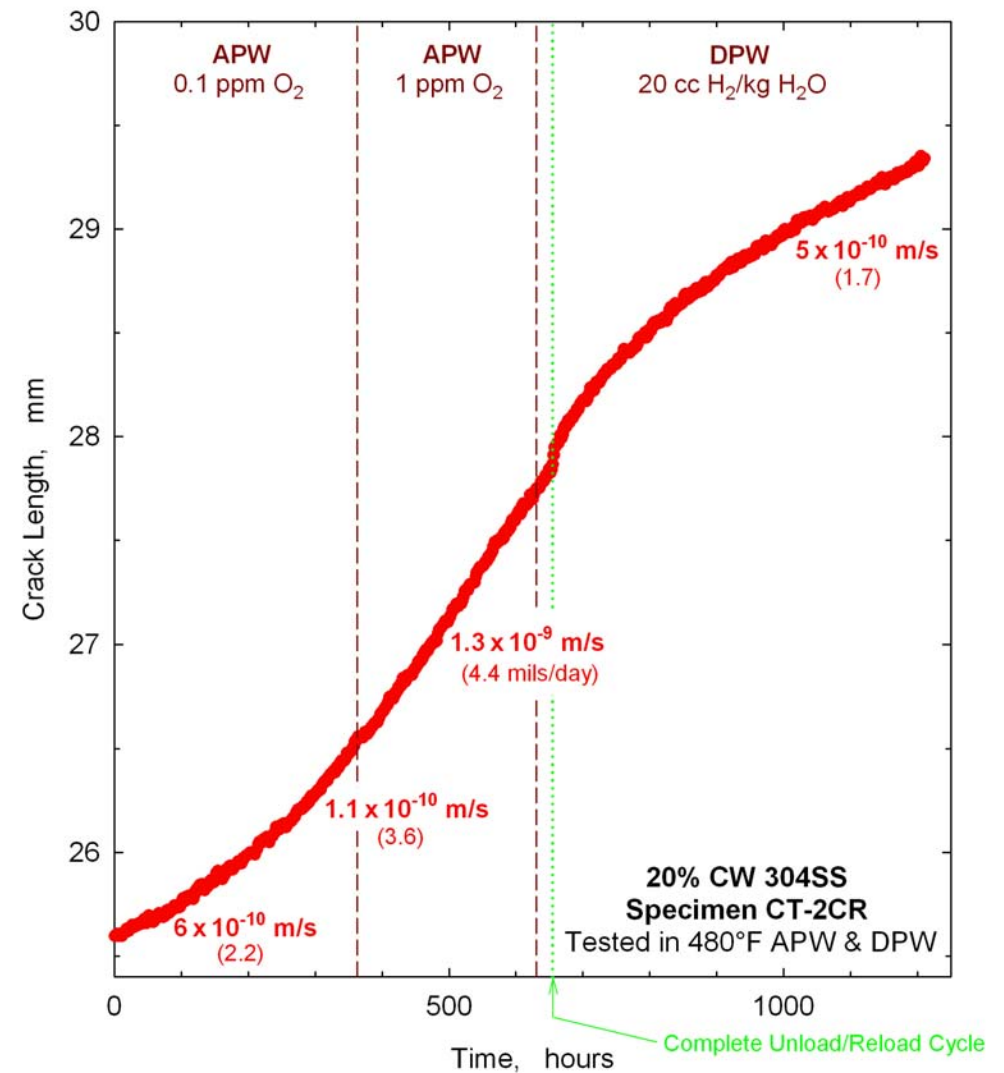
500s rise / 9000s hold / 500s fall

IGSCC occurred in

250°C APW with 0.1 ppm O₂

250°C APW with 1 ppm O₂

250°C DPW with 25 cc H₂/kg H₂O





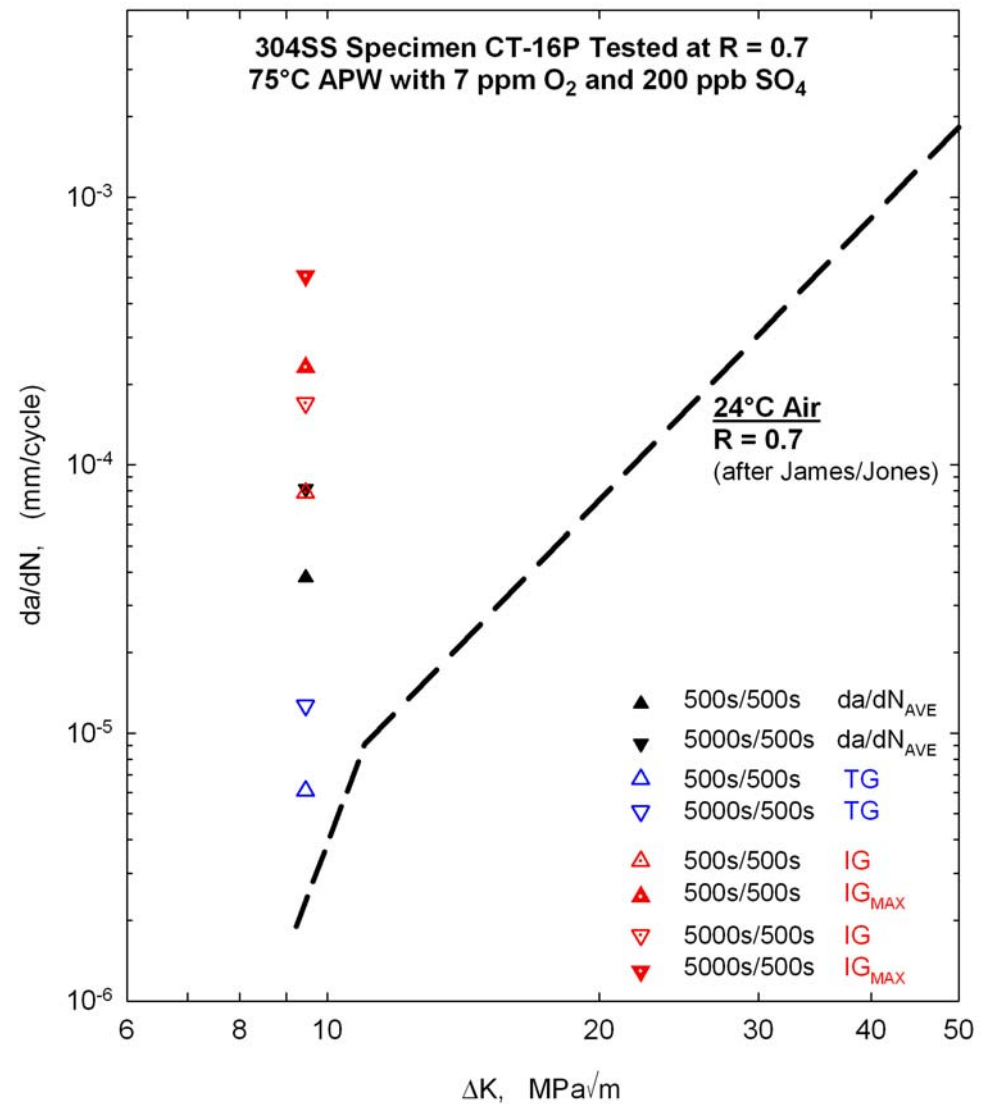
Fully Sensitized 304 SS
75°C APW with 200 ppb SO₄
Continuous Cycling at R = 0.7

In 75°C APW, da/dN_{AVE} is an order of magnitude greater than da/dN in RT air.

Increasing the rise time from 500s to 5000s doubles da/dN .

TG portion of the crack shows a modest (2 to 4-fold) acceleration in CGRs.

IG portion of the crack shows a 30 to 50-fold acceleration in CGRs.





304 SS tested at R = 0.7
250°C APW/DPW + 200 ppb SO₄
500s rise / 9000s hold / 500s fall

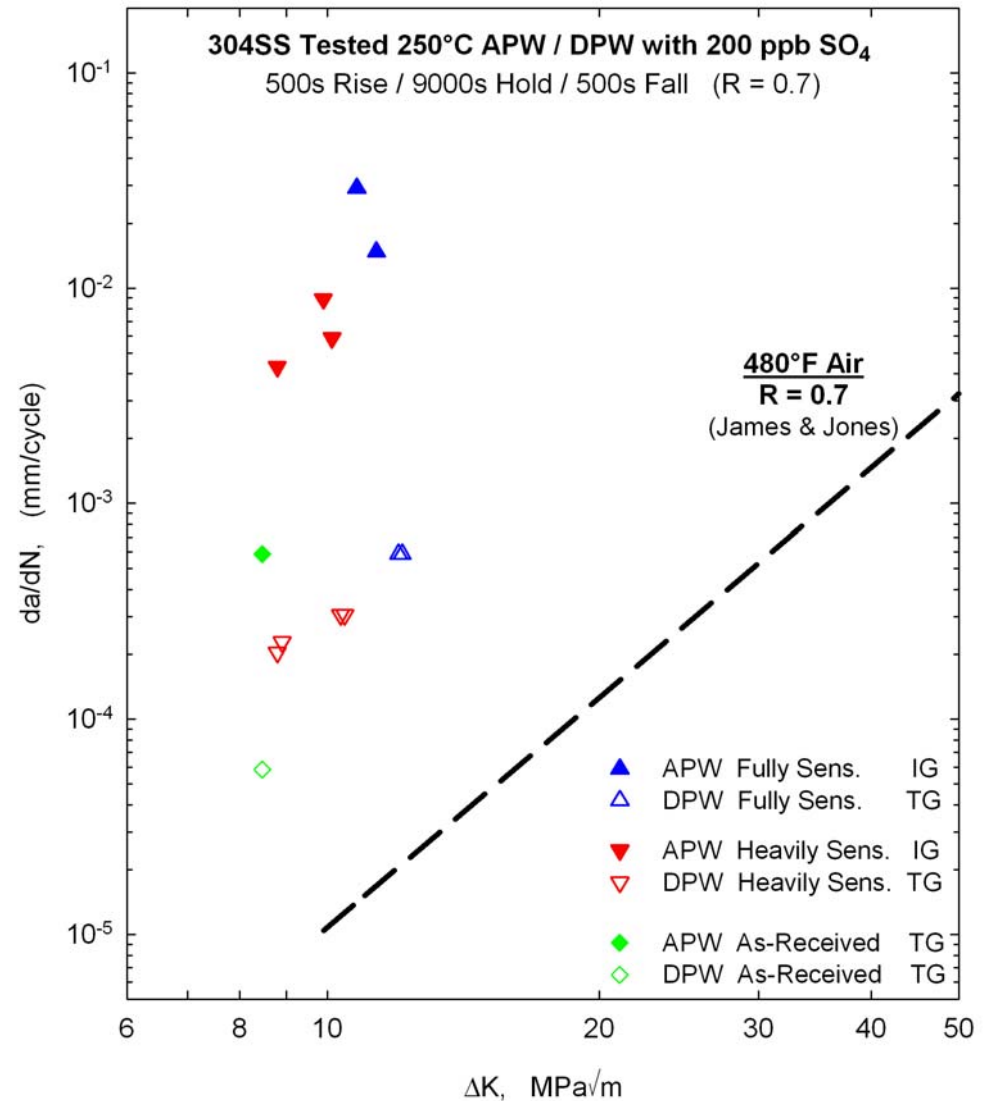
da/dN in 250°C DPW, where cracking mode is TG, is 10X to 30X greater than da/dN in air.

In 250°C APW, sensitized 304 SS exhibits exceptionally high CGRs due to IGSCC.

IGSCC rates in APW are 20X to 40X the TG-CGRs in DPW.

In 250°C APW, CGRs for as-received 304SS are 1/10th of those for sensitized SS.

Lower CGRs are expected for the non-sensitized material, because it resists IG cracking and fails by TG faceting.



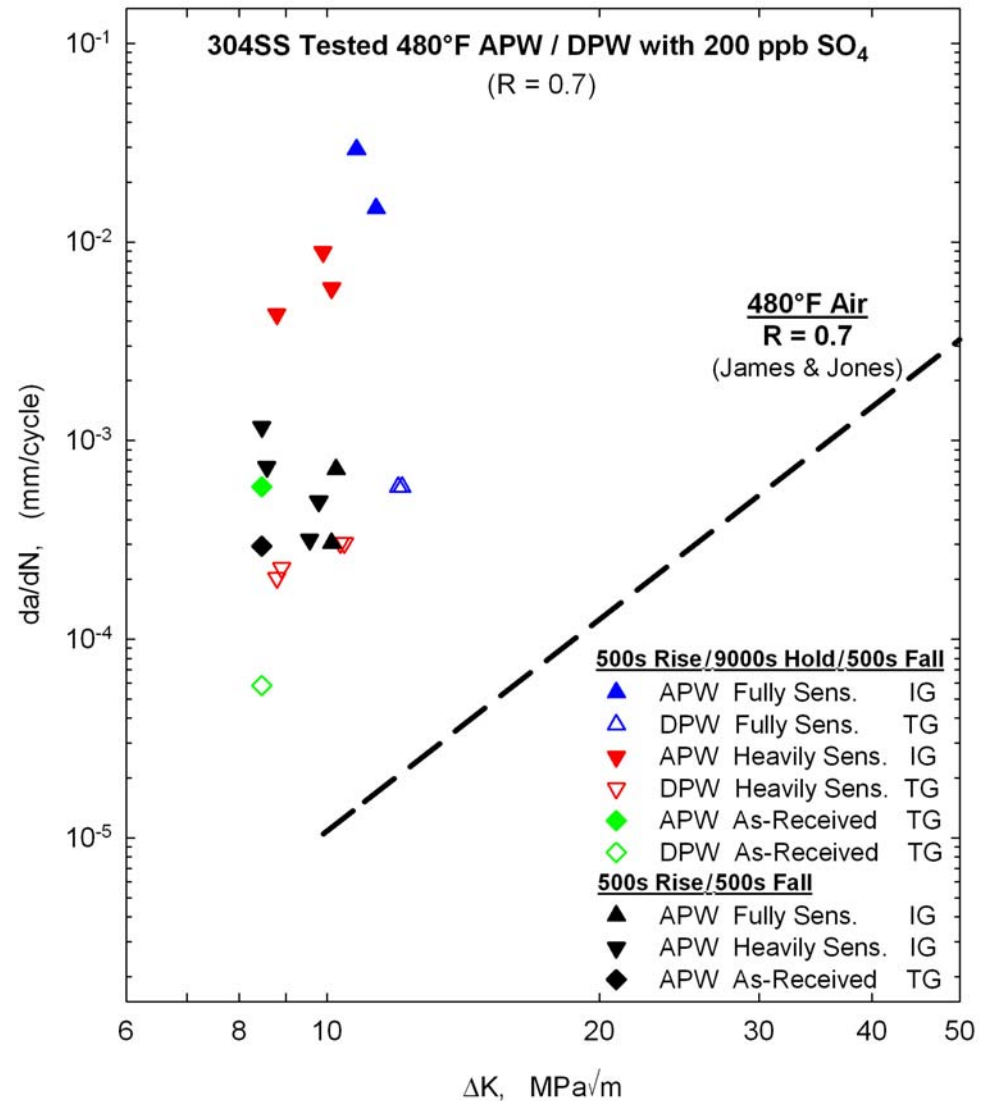


304 SS tested at $R = 0.7$ 250°C APW/DPW + 200 ppb SO_4

da/dN values generated under continuous cycling (500s / 500s) are an order of magnitude lower than da/dN values associated with IGSCC (9000s hold).

da/dN values obtained in 250°C APW under continuous cycling (500s rise / 500s fall) are slightly higher than rates for sensitized 304 SS tested in 250°C DPW with a 9000s hold.

Under continuous cycling, sensitized and non-sensitized 304 SS exhibited similar da/dN values, even though cracking modes were different.





20% Cold Rolled 304 SS
250°C APW/DPW
(R = 0.7)

Continuous cycling in 250°C APW with a 500s or 5000s rise time causes an order of magnitude increase in da/dN , relative to air rates. The cracking mode was **TG**.

The exceptionally high CGRs observed when a 9000s hold time was introduced was associated with an **IGSCC** mechanism.

IGSCC continued to occur in 250°C DPW. da/dN values in DPW were slightly lower than those in APW.

